# Supporting Information

## Organocatalytic Michael-Cyclization Cascade of 4-Oxa-α, β-Unsaturated Carboxylic Acids with Aldehydes: Facile Synthesis of Chiral γ-Lactols and Trisubstituted γ-Lactones

Jun-Bing Lin,[†,‡] Shi-Ming Xu,[†] Ji-Kang Xie,[†] Hong-Yu Li,[‡] and Peng-Fei Xu*[,†]

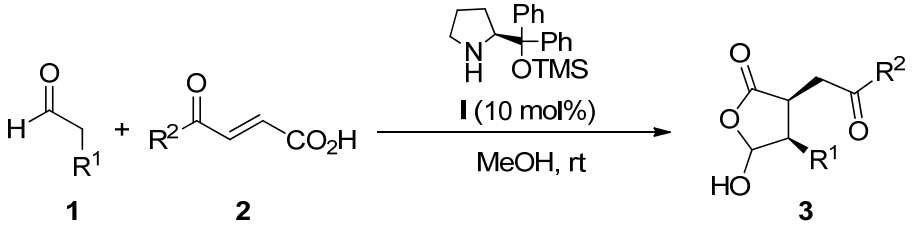
[†] *State Key Laboratory of Applied Organic Chemistry and College of Chemistry and Chemical Engeneering, Lanzhou University, Lanzhou 730000, China.* [‡] *Institute of Microbiology, School of Life Sciences, Lanzhou University, Lanzhou 730000, China*

xupf@lzu.edu.cn

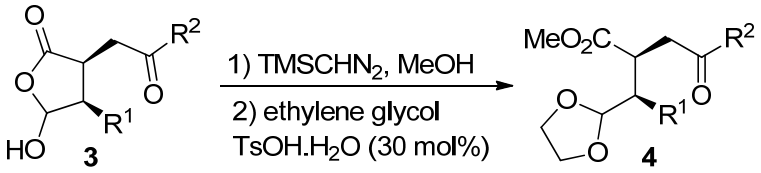
# 1.General Information

All reactions were carried out in glassware with magnetic stirring. Unless otherwise noted, solvents were either purchased from commercial suppliers or purified by standard procedures. Purification of the reaction products was carried out by flash column chromatography at reduced pressure. Thin layer chromatography was performed using silica gel plates and visualized with ultraviolet light. $^{1}H$ NMR spectra was recorded on Bruker 400 MHz spectrometer and the chemical shifts were reported in ppm (δ) relative to internal standard TMS (0 ppm). $^{13}C$ spectra are recorded at 100 MHz and referenced to the central $CDCl_3$ resonance (77.0 ppm). HRMS were made by Thermo Fisher mass instrument (Orbitrap Elite). IR spectra are recorded by Nicolet NEXUS 670 FT-IR instrument and are reported in wavenumbers ($cm^{-1}$). HPLC analyses were conducted on the Daicel Chiralpak AD-H column on Agilent 1100 series or IC colunm on Waters 2900/1525 series and eluting with MeOH/$CH_2Cl_2$/*n*-hexane. Optical rotations were recorded on a Perkin Elmer 341 polarimeter with $[\alpha]_D$ values reported in degrees; concentration (c) is in g/100 mL. Carboxylic acid[1] **2** and catalyst[2] **I** were synthesized according to the previously reported methods.

# 2. Synthesis and Characterization of **3** and **4**

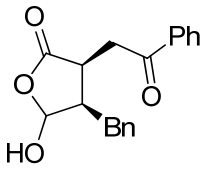



To a solution of catalyst **I** (0.02 mmol ) in MeOH (1 mL), unsaturated carboxylic acid **2** (0.2 mmol ) and aldehyde **1** (0.3 mmol) were added successively. The reaction was stirred and monitored by TLC. Upon the complete consumption of **2**, the solvent was evaporated and the residue was purified by flash column chromatography to afford γ-lactol **3**.

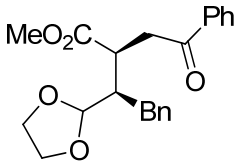



To a solution of γ-lactol **3** (0.2 mmol) in MeOH (2 mL), $TMSCHN_2$ (0.5 mL, 2.0 M in hexanes) was slowly added, and the resulting mixture was stirred for another 15 min and quenched by 1-2 drops of acetic acid. The solvent was evaporated in vacuo and the residue was purified by flash column chromatography affording α-stereogenic esters, which was dissolved in $CH_2Cl_2$ (2 mL) and treated with ethylene glycol (0.22 mmol) and then $TsOH.H_2O$ (12.0 mg). After the consumption of the starting material, the reaction was partitioned between $CH_2Cl_2$ and saturated $NaHCO_3$. The organic layer was separated, washed with brine, dried ($Na_2SO_4$), and evaporated. Flash

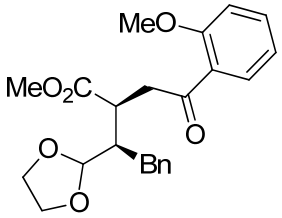
chromatography of the residue over silica gel gave **4** as colorless oil.




(3S,4R)-4-benzyl-5-hydroxy-3-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one(**3a**). white solid; 86% yield. $[\alpha]_D^{20}$ = 28.65, (*c* 1.01, THF); $^1$H NMR (400 MHz, acetone-d$^6$): δ 2.45 (s, 1H), 2.83 (dd, *J*= 4.8, 14.0Hz, 1H), 3.01-3.07 ( m, 1H), 3.42 (dd, *J*= 4.8, 8.4 Hz, 1H), 3.58 (dd, *J*= 3.2, 18.4 Hz, 1H), 3.77(s, 1H), 5.55(s, 1H), 7.18-7.22 (m, 1H), 7.26-7.32 ( m, 4H), 7.53-7.57 (m, 2H), 7.63-7.68 (m, 1H), 8.05-8.07 (m, 2H); $^{13}$C NMR (100 MHz, acetone-d$^6$): δ 32.9, 34.3, 37.7, 46.1, 99.7, 126.3, 128.0, 128.6, 128.7, 128.8, 133.2, 136.7, 138.8, 177.4, 197.1 ppm; HRMS calculated for $[C_{19}H_{18}O_4+H]^+$: 311.1278; found 311.1275.

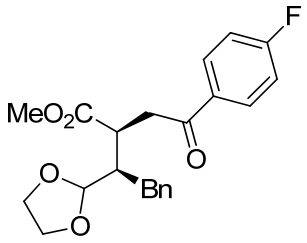



(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-oxo-4-phenylbutanoate(**4a**). colorless oil; 55% yield for both diastereomers from **3a**; 99% ee; $[\alpha]_D^{20}$ = 16.03, (*c* 1.06, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.67 (dd, *J*= 9.2,13.6 Hz,1H), 2.72-2.76 ( m, 1H), 2.99-3.08 ( m, 2H), 3.34-3.38(m, 1H), 3.65-3.72(m, 4H), 3.84-3.91(m, 2H), 3.96-4.03(m, 2H), 4.91(d, *J*= 3.2Hz,1H), 7.21-7.25 ( m, 3H), 7.28-7.33 ( m, 2H), 7.46-7.49 (m, 2H), 7.55-7.59 (m, 1H), 7.96-7.99 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 36.2, 39.2, 44.7, 51.7, 64.6, 65.1, 104.4, 126.2, 128.0, 128.4, 128.5, 129.0, 132.9, 136.8, 139.4, 174.7, 198.3 ppm; IR: ν 2951, 1778, 1733, 1686, 1599, 1451, 1206, 1152, 734 cm$^{-1}$, HRMS calculated for $[C_{22}H_{24}O_5+Na]^+$: 391.1516; found 391.1526. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 15.1 min, $t_{major}$= 18.3 min.

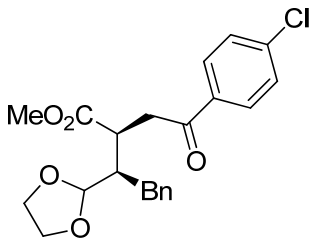



(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(2-methoxyphenyl)-4-oxobu tanoate (**4b**).colorless oil ; 37% yield for both diastereomers from **3b** ; 98% ee; $[\alpha]_D^{20}$ = 30.21, (*c* 0.96, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.61-2.71 (m, 2H), 2.95(dd, *J*=5.2, 13.2 Hz,1H), 3.18(dd, *J*=2.8, 18.4Hz,1H), 3.27-3.31(m, 2H), 3.53-3.62(m, 4H),

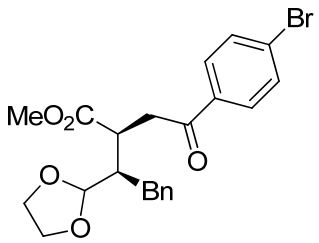
3.80-3.83(m, 2H), 3.85-3.95(m, 3H), 3.96-3.99(m, 2H), 4.91(d, *J*= 3.2Hz,1H), 6.94-7.00( m, 2H), 7.16-7.29 ( m, 5H), 7.42-7.46 (m, 1H), 7.67-7.69 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 39.3, 41.8, 45.1, 51.5, 55.5, 64.6, 65.1, 104.6, 111.5, 120.6, 126.1, 128.1, 128.4, 129.1, 130.3, 133.3, 139.7, 158.5, 174.9, 200.2 ppm; IR: ν 2949, 1777, 1734, 1677, 1602, 1468, 1205, 1153, 912, 733 cm$^{-1}$, HRMS calculated for $[C_{23}H_{26}O_6+Na]^+$: 421.1622; found 421.1625. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 20.1 min, $t_{major}$= 24.1 min.



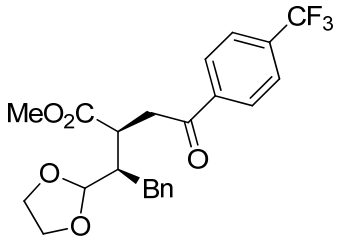

(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(4-fluorophenyl)-4-oxobuta noate (**4c**). colorless oil; 47% yield for both diastereomers from **3c**; 99% ee; $[\alpha]_D^{20}$ = 18.42, (*c* 1.14, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.62 (dd, *J*= 9.2, 13.6 Hz,1H), 2.69-2.74 (m, 1H), 2.94-3.00 (m, 2H), 3.30-3.34 (m, 1H), 3.58-3.65 (m, 4H), 3.80-3.88 (m, 2H), 3.91-4.00 (m, 2H), 4.87 (s, *J*= 3.6 Hz, 1H), 7.09-7.13 ( m, 2H), 7.20-7.31 ( m, 5H), 7.95-7.98 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 36.1, 39.3, 44.7, 51.7, 64.7, 65.2, 104.4, 115.4, 115.6, 126.3, 128.5, 129.0, 130 .6, 130.7, 133.2, 133.3, 139.3, 164.4, 166.9, 174.7, 196.8 ppm; IR: ν 2951, 1774, 1734, 1681, 1458, 1358, 1205, 1152, 1067, 911, 732 cm$^{-1}$, HRMS calculated for $[C_{22}H_{23}F_1O_5+Na]^+$: 409.1422; found 409.1432. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 15.9 min, $t_{major}$= 18.6 min.



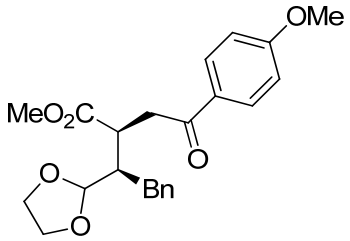

(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(4-chlorophenyl)-4-oxobuta noate(**4d**). colorless oil; 51% yield for both diastereomers from **3d**; 99% ee; $[\alpha]_D^{20}$ = 17.03, (*c* 1.35, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.59-2.74 ( m, 2H), 2.93-3.00 ( m, 2H), 3.29-3.33 (m, 1H), 3.56-3.65 ( m, 4H), 3.83-3.88 ( m, 2H), 3.94 -4.00 ( m, 2H), 4.87 (d, *J*= 3.6 Hz,1H), 7.19-7.22 ( m, 3H), 7.25-7.30 ( m, 2H, 7.41-7.43 ( m, 2H), 7.86 - 7.88 ( m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.4, 36.1, 39.3, 44.7, 51.8, 64.7, 65.2, 104.5, 126.3, 128.5, 128.7, 129.0, 129.5, 135 .2, 139.3, 139.4, 174.6, 197.2 ppm; IR: ν 2950, 1777, 1733, 1687, 1598, 1457, 1206, 1154, 911, 733 cm$^{-1}$, HRMS calculated for $[C_{22}H_{23}ClO_5+Na]^+$: 425.1126; found 425.1138. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 17.8 min, $t_{major}$= 22.8 min.




(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(4-bromophenyl)-4-oxobuta noate (**4e**).colorless oil; 49% yield for both diastereomers from **3e**; 99% ee; $[\alpha]_D^{20}$ =26.29, (*c* 0.95, $CH_2Cl_2$); $^1H$ NMR (400 MHz, $CDCl_3$): δ 2.61 (dd, *J*= 4.8, 9.2 Hz, 1H), 2.65-2.74 (m, 1H), 2.92-3.00 (m, 2H), 3.29-3.33 (m, 1H), 3.56-3.63 (m, 4H), 3.81-3.87 (m, 2H), 3.90-4.01 (m, 2H), 4.87(d, *J*= 3.6Hz, 1H), 7.19-7.21 ( m, 3H), 7.25-7.30 ( m, 3H), 7.57-7.59 (m, 2H), 7.78-7.80 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 33.3, 36.1, 39.2, 44.7, 51.7, 64.6, 65.1, 104.4, 126.3, 128.1, 128.5, 129.0, 129.5, 131.7, 135.5, 139.3, 174.6, 197.4 ppm; IR: ν 2948, 1777, 1673, 1598, 1483, 1287, 1152, 1067, 733 $cm^{-1}$, HRMS calculated for $[C_{22}H_{23}BrO_5+Na]^+$: 469.0621; found 469.0634. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 18.9 min, $t_{major}$= 25.1 min.

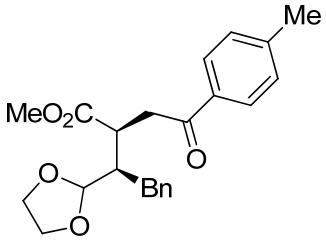



(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-oxo-4-(4-(trifluoromethyl)p henyl)butanoate (**4f**).colorless oil; 61% yield for both diastereomers from **3f**; 99% ee; $[\alpha]_D^{20}$ = 15.70, (*c* 1.21, $CH_2Cl_2$); $^1H$ NMR (400 MHz, $CDCl_3$): δ 2.63 (dd, *J*= 9.6, 13.6 Hz,1H), 2.71-2.77 ( m, 1H), 2.97-3.02 ( m, 2H), 3.31-3.35 ( m, 1H), 3.62-3.69 ( m, 4H), 3.82-3.88 ( m, 2H), 3.92-4.00 ( m, 2H), 4.88 (d, *J*= 3.6 Hz,1H), 7.20-7.31 ( m, 5H), 7.70-7.72 (m, 2H), 8.02-8.04 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 33.4, 36.5, 39.4, 44.6, 51.8, 64.7, 65.2, 104.5, 125.5, 125.6, 125.6, 126.3, 128.4, 128.5, 129.0, 134.1, 134.4, 139.2, 139.6, 174.5, 197.6 ppm; IR : ν 2948, 1778, 1735, 1687, 1593, 1206, 1067, 912, 820, 733 $cm^{-1}$, HRMS calculated for $[C_{23}H_{23}F_3O_5+Na]^+$: 459.1390; found 459.1407. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 15.1 min, $t_{major}$= 20.1 min.

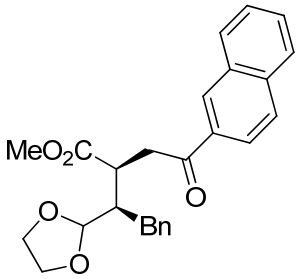



(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(4-methoxyphenyl)-4-oxobu tanoate (**4g**).colorless oil; 40% yield for both diastereomers from **3g**; 99% ee; $[\alpha]_D^{20}$ =12.84, (*c* 1.09, $CH_2Cl_2$); $^1H$ NMR (400 MHz, $CDCl_3$): δ 2.61-2.72 (m, 2H), 2.95-3.01 (m, 2H), 3.30-3.35 (m, 1H), 3.56-3.62 (m, 4H), 3.82-3.88 (m, 5H),

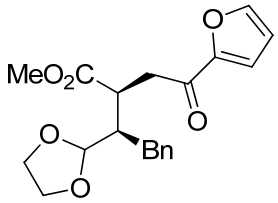
3.93-3.99 (m, 2H), 4.88 (dd, *J*= 3.6Hz,1H), 6.91-6.93 ( m, 2H), 7.20-7.21 ( m, 3H), 7.25-7.30 (m, 3H), 7.92-7.94 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 35.9, 39.2, 44.8, 51.7, 55.4, 64.7, 65.2, 104.5, 113.5, 126.2, 128.4, 129.1, 129.9, 130.3, 139.5, 163.3, 174.8, 196.8 ppm; IR : ν 2952, 1777, 1735, 1692, 1601, 1457, 1324, 1153, 1067, 911, 734 cm$^{-1}$, HRMS calculated for $[C_{23}H_{26}O_6+Na]^+$: 421.1622; found 421.1634. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 29.1 min, $t_{major}$= 41.5 min.



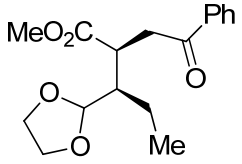

(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-oxo-4-(p-tolyl)butanoate (**4h**).colorless oil; 66% yield for both diastereomers from **3h**; 99% ee; $[\alpha]_D^{20}$ =13.58, (*c* 0.95, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.41 (s, 3H), 2.61-2.72 (m, 2H), 2.95-3.03 (m, 2H), 3.30-3.34 (m, 1H), 3.58-3.65 (m, 4H), 3.81-3.88 (m, 2H), 3.91-4.00 (m, 2H), 4.88 (d, *J*= 3.6Hz,1H), 7.17-7.30 ( m, 7H), 7.83-7.85 ( m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 21.6, 33.3, 36.2, 39.2, 44.8, 51.7, 64.7, 65.2, 104.5, 126.2, 128.1, 128.5, 129.0, 129.1, 134.4, 139.5, 143.7, 174.8, 197.6 ppm; IR: ν 2950, 1777, 1734, 1682, 1605, 1456, 1205, 1152, 1067, 733 cm$^{-1}$, HRMS calculated for $[C_{23}H_{26}O_5+Na]^+$: 405.1672; found 405.1685. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 17.6 min, $t_{major}$= 25.4 min.



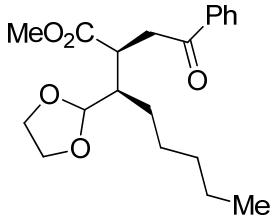

(S)-methyl2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(naphthalen-2-yl)-4-oxobuta noate (**4i**).colorless oil; 56% yield for both diastereomers from **3i**; 99% ee; $[\alpha]_D^{20}$ =19.17, (*c* 0.81, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.66-2.76 (m, 2H), 3.02 (dd, *J*= 4.4, 12.4 Hz,1H), 3.13 (dd, *J*= 3.2, 18.0 Hz, 1H), 3.38-3.43 (m, 1H), 3.64 (s, 3H), 3.74-3.81 (m, 1H), 3.85-3.88 (m, 2H), 3.90-4.03 (m, 2H), 4.93 (d, *J*= 3.2 Hz,1H), 7.19-7.32 ( m, 5H), 7.53-7.61 ( m, 2H), 7.86-7.89 (m, 2H), 7.96-8.01 (m, 2H), 8.45 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 36.5, 39.4, 44.9, 51.7, 64.7, 65.2, 104.6, 123.9, 126.3, 126.6, 127.7, 128.3, 128.4, 128.5, 129.1, 129.5, 129.6, 132.5, 134.2, 135.6, 139.5, 174.8, 198.2 ppm; IR: ν 2939, 1716, 1602, 1455, 1360, 1205, 1152, 1067, 700 cm$^{-1}$, HRMS calculated for $[C_{26}H_{26}O_5+Na]^+$: 441.1672; found 441.1685. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 26.8 min, $t_{major}$= 28.5 min.



(S)-methyl 2-((R)-1-(1,3-dioxolan-2-yl)-2-phenylethyl)-4-(furan-2-yl)-4-oxobutanoate (**4j**). colorless oil; 61% yield for both diastereomers from **3j**; 99% ee; $[\alpha]_D^{20}$ = 22.03, (*c* 0.95, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.62 (dd, *J*= 11.2, 13.6 Hz, 1H), 2.68-2.73 (m, 1H), 2.89-2.99 (m, 2H), 3.27-3.31 (m, 2H), 3.52 (dd, *J*= 10.8, 17.6 Hz,1H), 3.61(s, 3H), 4.86 (d, *J*=4.4 Hz, 1H), 6.51-6.52 (m, 1H), 7.17-7.21 ( m, 4H), 7.26-7.30 ( m, 2H), 7.57-7.58 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 33.3, 35.7, 38.7, 44.7, 51.7, 64.6, 65.1, 104.4, 112.1, 116.8, 126.3, 128.5, 129.0, 139.3, 146.1, 152.6, 174.5, 187.5 ppm; IR: ν 2952, 1733, 1689, 1587, 1455, 1399, 1206, 1152, 1069, 733 cm$^{-1}$, HRMS calculated for $[C_{20}H_{22}O_6+Na]^+$: 381.1309; found 381.1324. HPLC analysis: Chiralpak AD-H, *n*-hexane / *i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 15.8 min, $t_{major}$= 17.8 min.

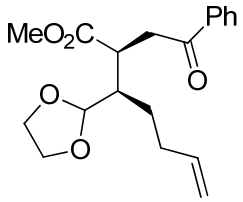


(2S,3R)-methyl3-(1,3-dioxolan-2-yl)-2-(2-oxo-2-phenylethyl)pentanoate(**4l**).
colorless oil; 54% yield for both diastereomers from **3l**; 99% ee;$[\alpha]_D^{20}$ = 48.27, (*c* 0.87, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 1.01 ( t, 3H), 1.33-1.44 ( m, 1H), 1.59-1.70 ( m, 1H), 2.23-2.29 ( m, 1H), 2.96 ( dd, *J* = 2.8 ,18.0 Hz,1H), 3.41-3.46 ( m, 1H), 3.64-3.75 ( m, 4H), 3.80-3.87 ( m, 2H), 3.90 - 3.98 ( m, 2H), 4.87 ( d, *J* = 4.0 Hz,1H), 7.44 - 7.48 ( m, 2H), 7.54 - 7.57 (m, 1H), 7.98-8.00 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$):
δ 12.1, 20.3, 35.9, 39.3, 44.7, 51.7, 64.6, 65.1, 105.2, 128.1, 128.4, 132.9, 136.9, 175.
1, 198.8 ppm; IR: ν 2931, 1777, 1734, 1688, 1599, 1459, 1206, 1153, 911, 733 cm$^{-1}$, HRMS calculated for $[C_{17}H_{22}O_5+Na]^+$: 329.1359; found 329.1362. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0mL/min, λ=254 nm, $t_{minor}$= 11.6 min, $t_{major}$= 13.1 min.



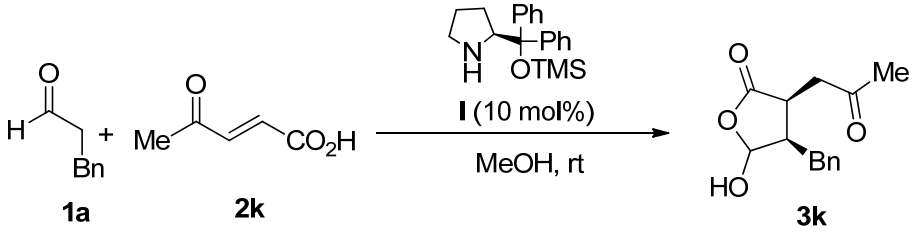
(2S,3R)-methyl 3-(1,3-dioxolan-2-yl)-2-(2-oxo-2-phenylethyl)octanoate(**4m**).
colorless oil; 39% yield for both diastereomers from **3m**; 97% ee; $[\alpha]_D^{20}$ = 28.57, (*c* 1.41, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 0.86-0.89 (m, 3H), 1.25-1.42 (m, 8H), 2.33-2.37 (m, 1H), 2.95 (dd, *J*=2.8, 18Hz, 1H), 3.39-3.43(m, 1H), 3.64-3.71(m, 4H), 3.80-3.87(m, 2H), 3.90-3.97(m, 2H), 4.86 (d, *J* = 4 Hz, 1H), 7.44-7.48 (m, 2H), 7.54-7.57 (m, 1H), 7.98-8.00 (m, 2H); $^{13}$C NMR (400 MHz, $CDCl_3$): δ 14.0,

22.5, 27.1, 27.2, 31.9, 35.9, 39.6, 42.9, 51.7, 64.6, 65.1, 105.3, 128.1, 128.4, 132.9, 13 6.9, 175.0, 198.8 ppm; IR: ν 2962, 1775, 1735, 1687, 1600, 1460, 1260, 1152, 1067, 756 $cm^{-1}$, HRMS calculated for $[C_{20}H_{28}O_5+Na]^+$: 371.1829; found 371.1841.
HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0mL/min, λ=254 nm, $t_{minor}$= 8.6 min, $t_{major}$= 9.4 min.

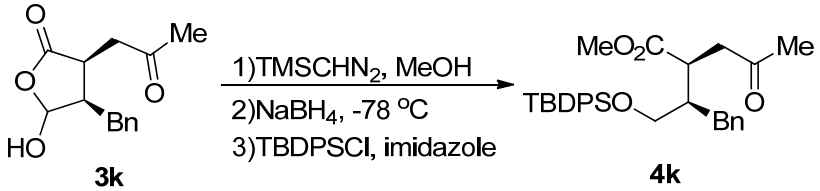



(2S,3R)-methyl 3-(1,3-dioxolan-2-yl)-2-(2-oxo-2-phenylethyl)hept-6-enoate (**4n**).
colorless oil; 29% yield for both diastereomers from **3n**; 95% ee; $[\alpha]_D^{20}$ = 40.63, (*c* 0.56, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 1.42-1.47 (m, 1H), 1.66-1.70 (m, 1H), 2.14-2.20 (m, 2H), 2.35-2.39(m, 1H), 2.96(dd, *J*=2.8, 18.0Hz, 1H), 3.40-3.43(m, 1H), 3.65-3.72(m, 4H), 3.81-3.88(m, 2H), 3.91-3.97(m, 2H), 4.87 (d, *J* = 4 Hz, 1H), 4.97-5.08 (m, 2H), 5.76-5.83(m, 1H), 7.44-7.48 (m, 2H), 7.54-7.58 (m, 1H), 7.98-8.00 (m, 2H); $^{13}$C NMR (400 MHz, $CDCl_3$): δ 26.4, 31.5, 36.0, 39.6, 42.2, 51.8, 64.6, 65.1, 105.1, 115.1, 128.1, 128.5, 133.0, 136.8, 138.0 , 174.8, 198.7 ppm; IR: ν 3361, 2926, 1734, 1686, 1654, 1449, 1228, 1162, 1115, 752, 697 $cm^{-1}$, HRMS calculated for $[C_{19}H_{24}O_5+Na]^+$: 355.1516; found 355.1521. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=90/10, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$= 10.6 min, $t_{major}$= 12.5 min.

synthesis of γ-lactol **3k** and its transformation to γ-keto ester **4k** [3]

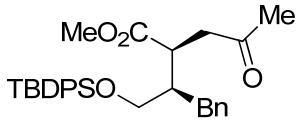



To a solution of catalyst **I** (0.02 mmol ) in MeOH (0.5 mL), unsaturated carboxylic acid **2k** (0.2 mmol ) and aldehyde **1a** (0.4 mmol) were added successively. The reaction was stirred for 48h and monitored by TLC. Upon the complete consumption of **2k**, the solvent was evaporated and the residue was subjected to flash column chromatography to afford both isomers of γ-lactol **3k**.




To a solution of γ-lactol **3k** (0.2 mmol) in MeOH (2 mL), $TMSCHN_2$ (0.8 mL, 2.0 M in hexanes) was slowly added, and the resulting mixture was stirred for another 15 min and quenched by 2-5 drops of acetic acid. The solvent was evaporated in vacuo and the residue was purified by flash column chromatography affording α-stereogenic

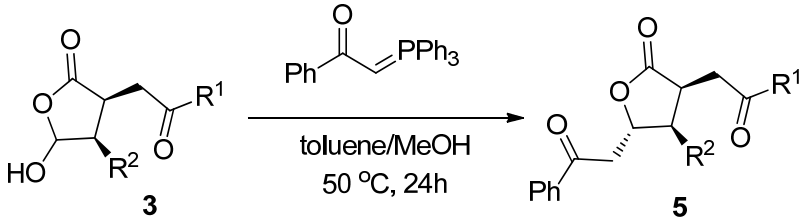
esters, which was then dissolved in mixed solvent of $CH_2Cl_2$ and EtOH (2 mL, EtOH/DCM=3:7). $NaBH_4$ (0.8 mmol) was added to the solution at -78 ºC. After 1h, the solution was treated with acetaldehyde (40 %, 1 mL), further stirred for 10 min and then warmed to rt. Then the solution was partitioned between ethyl acetate and water. The organic phase was separated and aqueous phase was extracted with ethyl acetate. The combined organic layers were dried over $Na_2SO_4$ and the solvent was evaporated and the residue was purified by flash column chromatography. The obtained semiacetal was dissolved in DMF at 0 ºC. Imidazole (10 eq.) and TBDPSCl (4 eq.) was added to this solution. After stirring at 0 ºC for 1h, the mixture was warmed to rt and monitored by TLC. Upon the totally conversion of the starting materials, the reaction was quenched with saturated $NaHCO_3$ and extracted with ethyl acetate. The combined organic phases were dried over $Na_2SO_4$ and concentrated. Flash chromatography of the residue over silica gel gave **4k** as a colorless oil.




(S)-methyl2-((R)-1-((tert-butyldiphenylsilyl)oxy)-3-phenylpropan-2-yl)-4-oxopentano ate (**4k**).

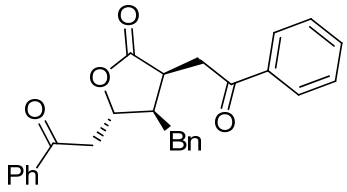
colorless oil; 31% yield for both diastereomers from **3k** ; 98% ee; $[\alpha]_D^{20}$ = 21.76, (*c* 0.87, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 1.06(s, 9H), 2.08(s, 3H), 2.21-2.26 (m, 1H), 2.35(dd, *J*=3.2, 17.6Hz, 1H), 2.53(dd, *J*=6.0, 13.6Hz, 1H), 2.73(dd, *J*=8.4, 13.6Hz, 1H), 2.92(dd, *J*=11.2, 17.6Hz, 1H), 3.11-3.16 (m, 1H), 3.49-3.51 (m, 2H), 3.61(s, 3H), 7.08-7.10(m, 2H), 7.17-7.25(m, 3H), 7.33-7.36(m, 4H), 7.39-7.43 (m, 2H), 7.57-7.60 (m, 4H); $^{13}$C NMR (400 MHz, $CDCl_3$): δ 19.2, 26.8, 30.0, 35.2, 41.1, 41.8, 44.0, 51.7, 62.8, 126.1, 127.6, 127.7, 128.3, 128.9, 129.6, 133.2, 133.3, 135.6, 135.6, 139.6, 175.1, 206.8 ppm; IR: ν 3492, 2931, 1719, 1471, 1428, 1361, 1163, 1112, 731, 702 $cm^{-1}$, HRMS calculated for $[C_{31}H_{38}O_4Si+Na]^+$: 525.2432; found 525.2427. HPLC analysis: Chiralpak IC Column, *n*-hexane/*i*-PrOH=99/1, flow rate 0.7 mL/min, λ = 229.7 nm, $t_{minor}$=25.9 min, $t_{major}$=23.5min.

## 3. Synthesis and Characterization of **5**

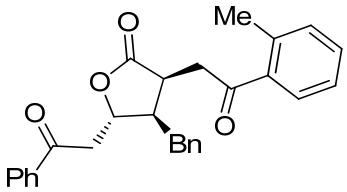



To a solution of γ-lactol **3** (0.2 mmol) in mixed solvent (2 mL, toluene/MeOH =3:1), triphenylphosphorane (0.6 mmol) was added to the reaction mixture and the solution was heated at 50 ºC for 24 h. Then the solvents were removed under reduced pressure and the residue was purified by flash column chromatography to afford the

trisubstituted γ-lactone **5** as white solid.

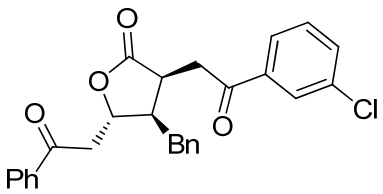



2,2'-((2**S**,3**R**,4**S**)-3-benzyl-5-oxotetrahydrofuran-2,4-diyl)bis(1-phenylethanone) (**5a**). white solid; 65% yield; 97% ee; $[\alpha]_D^{20}$ = -4.6, (*c* 1.74, $CH_2Cl_2$); mp 126-129 ºC;$^1$H NMR (400 MHz, $CDCl_3$): δ 2.51 (dd, *J* = 10.8, 13.6 Hz, 1H), 2.73 (dd, *J* = 5.6, 13.6 Hz, 1H), 3.02-3.09 (m, 2H), 3.32-3.41 (m, 2H), 3.55 (dd, *J* =3.6,18.8 Hz, 1H), 3.62-3.68 (m, 2H), 4.93-4.96 (m, 1H), 7.07-7.12 (m, 3H), 7.17-7.21 (m, 2H), 7.39-7.47 (m, 4H), 7.52-7.59 (m, 2H), 7.78-7.80 (m, 2H), 7.90-7.93 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.1, 35.2, 38.33, 42.7, 42.9, 78.9, 126.7, 128.0, 128.6, 128.7, 128.8, 133.5, 133.6, 135.9, 136.2, 137.6, 177.8, 195.6, 197.1 ppm; IR (KBr): ν 3438, 2912, 1764, 1683, 1449, 1327, 1212, 1147, 973, 758, 689 $cm^{-1}$, ESI-HRMS: calcd for $C_{27}H_{24}O_4$ $[M+H]^+$: 413.1747, Found 413.1738. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm, $t_{minor}$=6.4 min, $t_{major}$=7.2min.



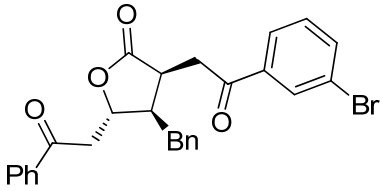

(3S,4R,5S)-4-benzyl-3-(2-oxo-2-(o-tolyl)ethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5b**).

white solid; 57% yield; 99% ee;$[\alpha]_D^{20}$ = -2.7, (*c*1.10, $CH_2Cl_2$); mp 115-118ºC;$^1$H NMR (400 MHz, $CDCl_3$): δ 2.49-2.55 (m,4H), 2.75 (dd, *J* = 6.0, 13.6 Hz, 1H), 3.00-3.09 (m, 2H), 3.27 (dd, *J* =9.6,18.4 Hz, 1H),3.38 (dd, *J* =7.2,16.8 Hz, 1H),3.51 (dd, *J* =3.6,18.4 Hz, 1H),3.63-3.68 (m, 2H), 4.92-4.96 (m, 1H), 7.12-7.18 (m, 3H), 7.20-7.28 (m, 4H), 7.38-7.44 (m, 3H), 7.53-7.57 (m, 1H), 7.65-7.67 (m, 1H), 7.79-7.80 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 21.6, 35.3, 37.5, 38.5, 42.6, 42.9, 78.8, 125.8, 126.7, 128.0, 128.6, 128.7, 128.8, 128.9, 132.0, 132.2, 133.4, 136.2, 136.3, 137.7, 138.7, 177.8, 195.6, 200.4 ppm;IR (KBr): ν 3437, 2917, 1763, 1685, 1449, 1412, 1324, 1213, 1147, 974, 764 $cm^{-1}$, ESI-HRMS: calcd for $C_{28}H_{26}O_4$ $[M+H]^+$: 427.1904, Found 427.1899. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 60/38/2, flow rate 1.0mL/min, λ=254 nm, $t_{minor}$=9.0 min, $t_{major}$=9.6 min.



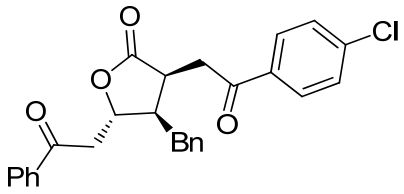
(3S,4R,5S)-4-benzyl-3-(2-(3-chlorophenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5c**).

white solid; 61% yield; 99% ee;$[\alpha]_D^{20}$ = -3.00, (*c* 0.80, $CH_2Cl_2$); mp 80-82°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.59 (dd, *J* = 9.6, 13.6 Hz, 1H), 2.72 (dd, *J* = 6.8, 13.6 Hz, 1H),3.03-3.14(m, 2H), 3.30 (dd, *J* =10.0,18.8 Hz, 1H), 3.38-3.52 (m, 2H), 3.61-3.67 (m, 1H), 4.93-4.96 (m, 1H), 7.08-7.11 (m, 3H), 7.16-7.20 (m, 2H), 7.37-7.45 (m, 3H), 7.53-7.58 (m, 1H), 7.74-7.76 (m, 1H), 7.80-7.83 (m, 2H);; $^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.2, 35.4, 38.2, 42.6, 42.7, 79.1, 126.0, 126.6, 128.0, 128.1, 128.6, 128.7, 128.8, 129.9, 133.4, 133.5, 134.9, 136.1, 137.3, 137.5, 177.6, 195.7, 195.8 ppm;IR (KBr): ν 3435, 2913, 1766, 1685, 1415, 1326, 1210, 1145, 972, 702cm$^{-1}$, ESI-HRMS: calcd for $C_{27}H_{23}ClO_4$ $[M+H]^+$: 447.1358, Found 447.1351. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0 mL/min, λ=254 nm, $t_{minor}$=6.7 min, $t_{major}$=7.8min.



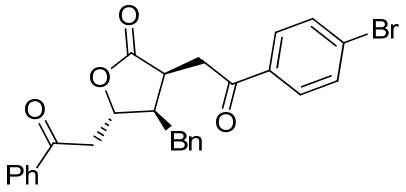
(3S,4R,5S)-4-benzyl-3-(2-(3-bromophenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5d**).

white solid; 69% yield; 99% ee;$[\alpha]_D^{20}$ = -1.9, (*c*1.04, $CH_2Cl_2$); mp 90-94°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.59 (dd, *J* = 9.6, 13.6 Hz, 1H), 2.71 (dd, *J* = 6.4, 13.6 Hz, 1H), 3.03-3.15(m, 2H), 3.30 (dd, *J* =10.0,18.8 Hz, 1H), 3.40-3.52 (m, 2H), 3.61-3.67 (m, 1H), 4.93-4.96 (m, 1H), 7.06-7.20 (m, 5H), 7.31-7.35 (m, 1H), 7.42-7.46 (m, 2H), 7.55-7.59 (m, 1H), 7.68-7.70 (m, 1H), 7.79-7.83 (m, 3H), 7.98 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.3, 35.5, 38.2, 42.6, 42.6, 79.2, 123.0, 126.5, 126.7, 127.9, 128.0, 128.7, 128.8, 130.2, 131.1, 133.5, 136.2, 136.3,137.5, 137.6, 177.6, 195.7, 195.8 ppm;IR (KBr): ν 2912, 1766, 1684, 1566, 1449, 1414, 1326, 1210, 1145, 972, 690cm$^{-1}$,ESI-HRMS: calcd for $C_{27}H_{23}BrO_4$ $[M+H]^+$: 491.0852, Found 491.0842. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH=50/47.5/2.5, flow rate 1.0mL/min, λ=254 nm, $t_{minor}$=6.7 min, $t_{major}$=7.5min.



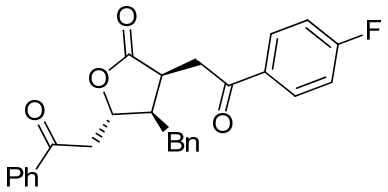
(3S,4R,5S)-4-benzyl-3-(2-(4-chlorophenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one(**5e**).

white solid; 70% yield;99% ee;$[\alpha]_D^{20}$ = -10.1, (*c*1.48, $CH_2Cl_2$); mp 112-115°C;$^1$H NMR (400 MHz, $CDCl_3$): δ2.57 (dd, *J* = 10.0, 14.0 Hz, 1H), 2.72 (dd, *J* = 6.4, 14.0 Hz, 1H), 3.03-3.13(m, 2H), 3.30 (dd, *J* =10.0,18.8 Hz, 1H), 3.40 (dd, *J* =7.2,16.8 Hz, 1H), 3.50 (dd, *J* =4.0,18.4 Hz, 1H), 3.61-3.66 (m, 1H), 4.93-4.96 (m, 1H), 7.09-7.11 (m, 3H), 7.16-7.20 (m, 2H), 7.41-7.45 (m, 4H), 7.54-7.58 (m, 1H), 7.80-7.83 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.1, 35.4, 38.2, 42.6, 42.7,79.1, 126.7, 128.0, 128.6, 128.7, 128.8, 128.9, 129.4, 133.5, 134.1, 136.2, 137.6, 140.0, 177.7, 195.7, 195.9 ppm;IR (KBr): ν 2922, 1761, 1687, 1590, 1323, 1212, 1145, 998, 971, 759, 742 689cm$^{-1}$,ESI-HRMS: calcd for $C_{27}H_{23}ClO_4$ $[M+H]^+$: 447.1358, Found 447.1352. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min, λ=254 nm, $t_{minor}$=6.6 min, $t_{major}$=7.5min.




(3S,4R,5S)-4-benzyl-3-(2-(4-bromophenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5f**).

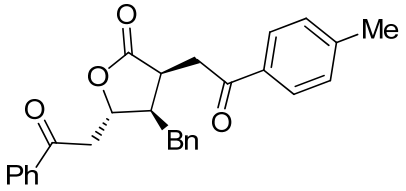
white solid; 65% yield;99% ee;$[\alpha]_D^{20}$ = -10.00, (*c*2.01, $CH_2Cl_2$); mp 132-135°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.57 (dd, *J* = 10.0, 14.0 Hz, 1H), 2.72 (dd, *J* = 6.4, 14.0 Hz, 1H), 3.03-3.13(m, 2H), 3.29 (dd, *J* =10.0,18.8 Hz, 1H), 3.40 (dd, *J* =6.8,16.8 Hz, 1H), 3.49 (dd, *J* =3.6,18.8 Hz, 1H), 3.61-3.66 (m, 1H), 4.93-4.96 (m, 1H), 7.09-7.11 (m, 3H), 7.16-7.20 (m, 2H), 7.41-7.45 (m, 2H), 7.55-7.60 (m, 3H), 7.74-7.76 (m, 2H), 7.80-7.82 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.1, 35.4, 38.2, 42.6, 42.7, 79.1, 126.7, 128.0, 128.7, 128.7, 128.8, 128.8, 129.5, 131.9, 133.5, 134.5, 136.2, 137.6, 177.7, 195.7, 196.1 ppm;IR (KBr): ν 3434, 2923, 1760, 1687, 1585, 1323, 1211, 1144, 996, 970, 759, 688cm$^{-1}$,ESI-HRMS: calcd for $C_{27}H_{23}BrO_4$ $[M+H]^+$: 491.0852, Found 491.0846. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/45/5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=7.3 min,$t_{major}$=8.2min.




(3S,4R,5S)-4-benzyl-3-(2-(4-fluorophenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5g**).

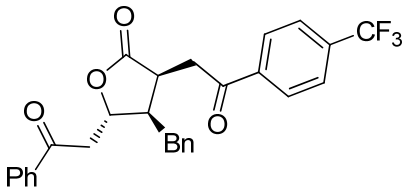
white solid; 61% yield; 99% ee;$[\alpha]_D^{20}$ = -28.7, (*c* 0.80, $CH_2Cl_2$); mp 146-149°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.56 (dd, *J* = 10.0, 13.6 Hz, 1H), 2.73 (dd, *J* = 6.0, 13.6 Hz, 1H), 3.04-3.13(m, 2H), 3.27-3.43 (m, 2H), 3.49 (dd, *J* =3.6,18.8 Hz, 1H),

3.61-3.67 (m, 1H), 4.93-4.7 (m, 1H), 7.07-7.20 (m, 7H),7.41-7.45 (m, 2H), 7.54-7.58 (m, 1H), 7.80-7.82(m, 2H), 7.91-7.94 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.0, 35.4, 38.3, 42.6, 42.7, 79.1, 115.6, 115.8, 126.7, 128.0, 128.1, 128.8, 130.6, 130.7, 132.3, 132.3, 133.5, 136.2, 137.6, 164.7, 167.2, 177.8, 195.5, 195.7 ppm;$^{19}$F NMR (376Hz, $CDCl_3$): δ –104.1 ppm;IR (KBr): ν 2909, 1769, 1680, 1595, 1505, 1412, 1353, 1230, 1139, 1001, 816, 736cm$^{-1}$,ESI-HRMS: calcd for $C_{27}H_{23}FO_4$ $[M+H]^+$: 431.1653, Found 431.1644. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min, λ=254 nm,$t_{minor}$=6.4 min,$t_{major}$=7.3min.




(3S,4R,5S)-4-benzyl-3-(2-oxo-2-(p-tolyl)ethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5h**).

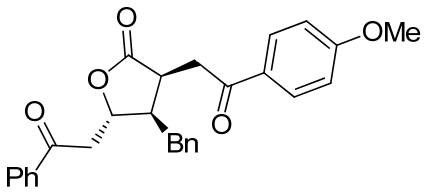
white solid; 71% yield; 99% ee;$[\alpha]_D^{20}$ = -123.5, (*c* 0.85, $CH_2Cl_2$); mp 113-116ºC; $^1$H NMR (400 MHz, $CDCl_3$): δ2.40 (s, 3H), 2.47-2.51 (m,1H), 2.72 (dd, *J* = 5.6, 13.6 Hz, 1H), 3.01-3.06(m, 2H), 3.29-3.40 (m, 2H), 3.52 (dd, *J* =3.6,18.8 Hz, 1H), 3.61-3.66 (m, 1H), 4.92-4.95 (m, 1H), 7.10-7.12 (m, 3H), 7.18-7.25 (m, 4H), 7.38-7.42 (m, 2H), 7.51-7.55 (m, 1H), 7.77-7.84(m, 4H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ21.5, 34.9, 35.0, 38.3, 42.6, 42.9, 78.7, 126.6, 127.9, 128.1, 128.5, 128.6, 128.8, 129.2, 133.3, 133.4, 136.1, 137.7, 144.4, 177.9, 195.6, 196.6 ppm;IR (KBr): ν 3431, 2921, 1770, 1677, 1604, 1323, 1208, 1176, 1141, 976, 700cm$^{-1}$,ESI-HRMS: calcd for $C_{28}H_{26}O_4$ $[M+H]^+$: 427.1904, Found 427.1898. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm, $t_{minor}$=6.3 min,$t_{major}$=7.4min.




(3S,4R,5S)-4-benzyl-3-(2-oxo-2-(4-(trifluoromethyl)phenyl)ethyl)-5-(2-oxo-2-phenyl ethyl)dihydrofuran-2(3H)-one(**5i**).

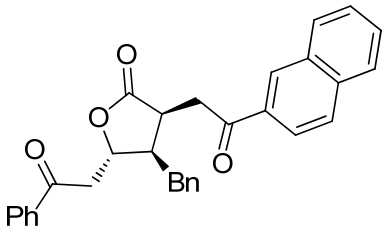
white solid; 57% yield; 99% ee;$[\alpha]_D^{20}$ = -5.0, (*c*1.00, $CH_2Cl_2$); mp 115-118ºC; $^1$H NMR (400 MHz, $CDCl_3$): δ2.58-2.75 (m, 2H), 3.05-3.15(m, 2H), 3.31-3.44 (m, 2H), 3.52 (dd, *J* =3.6,18.8 Hz, 1H), 3.64-3.69 (m, 1H), 4.93-4.97 (m, 1H), 7.03-7.17 (m, 5H), 7.39-7.43 (m, 2H), 7.52-7.56 (m, 1H), 7.67-7.69(m, 2H), 7.80-7.82 (m, 2H), 7.95-7.97 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.4, 35.4, 38.1, 42.5, 42.5, 79.2, 124.7, 125.2, 125.5, 125.6, 125.6, 126.6, 127.4, 127.8, 127.9, 128.3, 128.5, 128.6, 128.6, 133.4, 134.1, 134.4, 135.0, 136.1, 137.8, 138.4ppm;$^{19}$F NMR(376 Hz, $CDCl_3$): δ -63.1;IR (KBr): ν 2923, 2853, 1763, 1688, 1451, 1410, 1326, 1260, 1163, 1065, 805, 702cm$^{-1}$, ESI-HRMS: calcd for $C_{28}H_{23}F_3O_4$ $[M+H]^+$: 481.1621, Found 481.1616.

HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH=50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm, $t_{minor}$=6.1 min,$t_{major}$=7.1min.



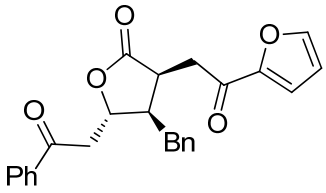

(3S,4R,5S)-4-benzyl-3-(2-(4-methoxyphenyl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5j**).

white solid; 49% yield; 99% ee;$[\alpha]_D^{20}$ = -28.8, (*c*1.10, $CH_2Cl_2$); mp 155-158°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.48 (dd, *J* = 10.8, 13.6 Hz, 1H), 2.74 (dd, *J* = 5.6, 14.0 Hz, 1H), 3.02-3.07(m, 2H), 3.28-3.40 (m, 2H), 3.51 (dd, *J* =3.6,18.8 Hz, 1H), 3.61-3.66 (m, 1H), 3.87 (s, 3H),4.92-4.96 (m, 1H), 6.92-6.94 (m, 2H),7.12-7.13 (m, 3H), 7.19-7.26 (m, 2H), 7.40-7.44 (m, 2H), 7.53-7.57 (m, 1H), 7.78-7.80(m, 2H), 7.90-7.93 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 34.7, 35.1, 38.4, 42.7, 43.0, 55.5, 78.7, 113.8, 126.6, 128.0, 128.6, 128.6, 128.8, 129.0, 130.3, 133.4, 136.2, 137.7, 163.8, 178.0, 193.5, 195.6 ppm;IR (KBr): ν 3435, 2924, 1774, 1678, 1601, 1416, 1327, 1259, 1174, 1027, 689cm$^{-1}$,ESI-HRMS: calcd for $C_{28}H_{26}O_5$ $[M+H]^+$: 443.1853, Found 443.1856. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=7.8 min,$t_{major}$=8.8min.



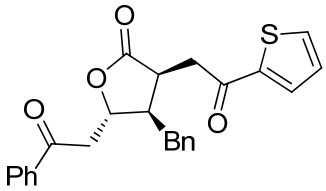

(3S,4R,5S)-4-benzyl-3-(2-(naphthalen-2-yl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one (**5k**).

white solid; 50% yield; 98% ee;$[\alpha]_D^{20}$ = -8.2, (*c*1.10, $CH_2Cl_2$); mp 158-161°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.56 (dd, *J* = 10.4, 14.0 Hz, 1H), 2.78 (dd, *J* = 6.0, 13.6 Hz, 1H), 3.07-3.13(m, 2H), 3.38-3.54 (m, 2H), 3.67-3.73 (m, 2H), 4.96-4.99 (m, 1H), 7.04-7.08 (m, 1H), 7.12-7.20 (m, 4H), 7.41-7.45 (m, 2H), 7.54-7.64 (m,3H), 7.80-7.82(m, 2H), 7.87-7.90 (m, 2H),7.95-7.99 (m, 2H), 8.44 (s, 1H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.2, 35.3, 38.4, 42.7, 42.9, 78.9, 123.5, 126.7, 126.9, 127.7, 128.0, 128.5, 128.7, 128.7, 128.8, 128.9, 129.6, 130.0, 132.4, 133.2, 133.5, 135.8, 136.2, 137.7, 177.9, 195.7, 197.0 ppm;IR (KBr): ν 3434, 2924, 1769, 1677, 1449, 1322, 1211, 1139, 974, 750, 690cm$^{-1}$,ESI-HRMS: calcd for $C_{31}H_{26}O_4$ $[M+H]^+$: 463.1904, Found 463.1906. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=7.2 min,$t_{major}$=7.8min.



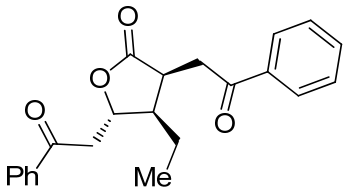
(3S,4R,5S)-4-benzyl-3-(2-(furan-2-yl)-2-oxoethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one(**5l**).

white solid; 59% yield; 99% ee;$[\alpha]_D^{20}$ = -15.0, (*c* 0.40, $CH_2Cl_2$); mp 138-140 °C;$^1$H NMR (400 MHz, $CDCl_3$): δ2.52 (dd, *J* = 10.8, 13.6 Hz, 1H), 2.79 (dd, *J* = 5.6, 13.6 Hz, 1H), 2.96-3.06(m, 2H), 3.21 (dd, *J* =9.6,18.4 Hz, 1H), 3.33-3.43 (m, 2H),3.60-3.65 (m, 1H), 4.91-4.95 (m, 1H), 6.52-7.54 (m, 1H), 7.11-7.14 (m, 3H), 7.18-7.26 (m, 3H), 7.39-7.43 (m, 2H), 7.52-7.58(m, 2H), 7.77-7.79 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 34.6, 35.1, 37.8, 42.6, 42.8, 78.7, 112.3, 117.5, 126.6, 127.9, 128.6, 128.8, 133.4, 136.1, 137.6, 146.6, 151.8, 177.5, 185.9, 195.6 ppm;IR (KBr): ν 3430, 2924, 1766, 1688, 1666, 1467, 1402, 1329, 1204, 1170, 1153, 1044, 994, 973, 759 cm$^{-1}$,ESI-HRMS: calcd for $C_{22}H_{25}O_5$ $[M+H]^+$: 403.1540, Found 403.1546. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=8.8 min,$t_{major}$=9.6min.



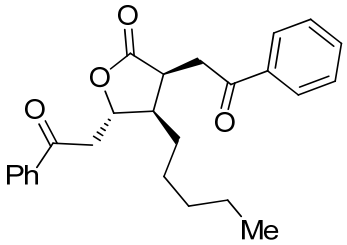

(3S,4R,5S)-4-benzyl-3-(2-oxo-2-(thiophen-2-yl)ethyl)-5-(2-oxo-2-phenylethyl)dihydrofuran-2(3H)-one(**5m**).

white solid; 65% yield; 99% ee;$[\alpha]_D^{20}$ = -4.0, (*c*1.00, $CH_2Cl_2$); mp 142-146°C; $^1$H NMR (400 MHz, $CDCl_3$): δ2.53 (dd, *J* = 10.4, 13.6 Hz, 1H), 2.80 (dd, *J* = 5.6, 13.6 Hz, 1H), 3.00-3.08(m, 2H), 3.25-3.40 (m, 2H),3.50 (dd, *J* =3.6,18.4 Hz, 1H), 3.61-3.66 (m, 1H), 4.92-4.96 (m, 1H), 7.11-7.14 (m, 4H), 7.20-7.25 (m, 2H), 7.40-7.44 (m, 2H), 7.53-7.55(m, 1H),7.65-7.70 (m, 2H), 7.78-7.80 (m, 2H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 35.2, 35.5, 38.3, 42.6, 42.8, 78.8, 126.7, 128.0, 128.2, 128.6, 128.7, 128.8, 132.3, 133.4, 134.1, 136.2, 137.6, 142.9, 177.5, 189.8, 195.6 ppm;IR (KBr): ν 2920, 1766, 1659, 1415, 1325, 1213, 1145, 973, 729, 688 cm$^{-1}$,ESI-HRMS: calcd for $C_{25}H_{22}O_4S$ $[M+H]^+$: 419.1312, Found 419.1315. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=7.8 min,$t_{major}$=8.4min.

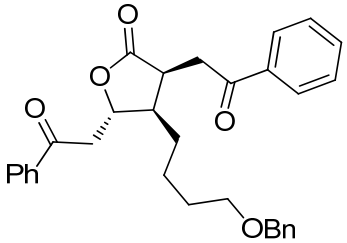



2,2'-((2S,3R,4S)-3-ethyl-5-oxotetrahydrofuran-2,4-diyl)bis(1-phenylethanone)(**5o**).white solid; 56% yield; 98% ee;$[\alpha]_D^{20}$ = -22.9, (*c* 0.61, $CH_2Cl_2$);mp 101-104°C; $^1$H NMR (400 MHz, $CDCl_3$): δ 1.01 (t, *J*=7.2 Hz, 3H), 1.22-1.33 (m, 1H), 1.37-1.46(m, 1H),2.52-2.58 (m, 1H), 3.25 (dd, *J* = 10.4, 19.2 Hz, 1H), 3,38 (dd, *J* = 8.0, 17.2 Hz, 1H), 3.52-3.57(m, 3H), 5.04-5.07 (m, 1H), 7.47-7.51 (m, 4H), 7.58-7.62 (m, 2H), 7.95-8.00 (m, 4H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 11.1, 21.5, 34.6, 37.6, 42.4, 43.1,

78.6, 128.0, 128.1, 128.7, 128.8, 133.6, 133.7, 136.0, 136.3, 178.1, 196.1, 197.2 ppm;IR (KBr): ν 3355, 2921, 1769, 1685, 1596, 1448, 1242, 1155, 1001, 756, 689cm$^{-1}$,ESI-HRMS: calcd for $C_{22}H_{24}O_4$ $[M+H]^+$: 351.1591, Found 351.1593.HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=5.7 min,$t_{major}$=6.3min.



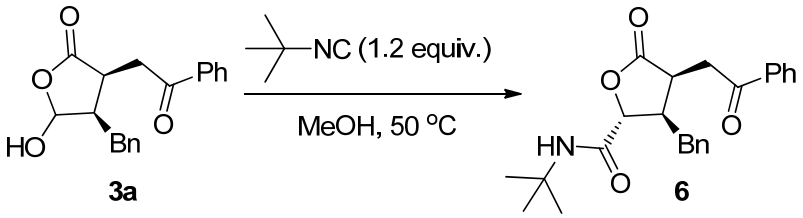

2,2'-((2S,3R,4S)-5-oxo-3-pentyltetrahydrofuran-2,4-diyl)bis(1-phenylethanone)(**5p**).white solid; 60% yield; 99% ee;$[\alpha]_D^{20}$ = -12.2, (*c* 0.91, $CH_2Cl_2$); mp 118-120 °C; $^1$H NMR (400 MHz, $CDCl_3$): δ 0.84 (t, *J*=6.8 Hz, 3H), 1.21-1.30(m, 8H), 2.57-2.63(m, 1H), 3.24 (dd, *J* = 10.8, 19.2 Hz, 1H), 3,36 (dd, *J* = 7.8, 17.2 Hz, 1H), 3.51-3.57 (m, 3H), 5.03-5.06 (m, 1H), 7.47-7.52 (m, 4H), 7.59-7.62 (m, 2H), 7.94-8.00 (m, 4H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 13.9, 22.3, 26.1, 28.6, 31.7, 34.7, 37.8, 41.8, 42.4, 79.0, 128.0, 128.7, 128.8, 133.6, 133.7, 136.1, 136.4, 178.1, 196.1, 197.2 ppm;IR (KBr): ν 2949, 2862, 1764, 1685, 1595, 1449, 1416, 1325, 1211, 1153, 970, 756, 688 cm$^{-1}$, ESI-HRMS: calcd for $C_{25}H_{28}O_4$ $[M+H]^+$: 393.2060, Found 393.2067. HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=5.5 min,$t_{major}$=5.8min.




2,2'-((2S,3R,4S)-3-(4-(benzyloxy)butyl)-5-oxotetrahydrofuran-2,4-diyl)bis(1-phenylethanone) (**5q**).

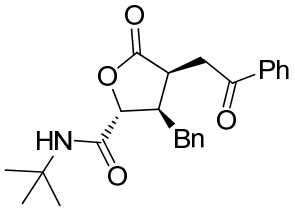
white solid; 51% yield; 99% ee; $[\alpha]_D^{20}$ = -6.0, (*c*1.00, $CH_2Cl_2$); mp 108-110 °C; $^1$H NMR (400 MHz, $CDCl_3$): δ 1.25-1.36 (m, 3H), 1.39-1.48(m, 2H),1.50-1.63 (m, 4H),2.58-2.64 (m, 1H),3.23 (dd, *J* = 10.4, 19.2 Hz, 1H), 3.33-3.43(m, 3H), 3.50-3.57 (m, 3H), 4.43 (s, 2H), 5.02-5.05 (m, 1H), 7.23-7.33 (m, 5H),7.45-7.51 (m, 4H), 7.57-7.62 (m, 2H), 7.94-7.98 (m, 4H);$^{13}$C NMR (100 MHz, $CDCl_3$): δ 23.2, 28.4, 29.6, 34.7, 37.7, 41.1, 42.3, 69.7, 72.8, 79.0, 127.5, 127.6, 128.0, 128.1, 128.3, 128.8, 133.6, 133.7, 136.1, 136.4, 138.5, 178.1, 196.1, 197.2 ppm;IR (KBr): ν 3435, 2955, 1770, 1681, 1595, 1450, 1365, 1261, 1227, 1153, 1105, 1025, 975, 799, 689 cm$^{-1}$,ESI-HRMS: calcd for $C_{31}H_{32}O_5$ $[M+H]^+$: 485.2322, Found 485.2329.HPLC analysis: Chiralpak IC, *n*-hexane/$CH_2Cl_2$/MeOH= 50/47.5/2.5, flow rate 1.0mL/min,λ=254 nm,$t_{minor}$=5.5min, $t_{major}$=7.9min.

## 4. Synthetical Transformations of **3a** to **6** and **7**

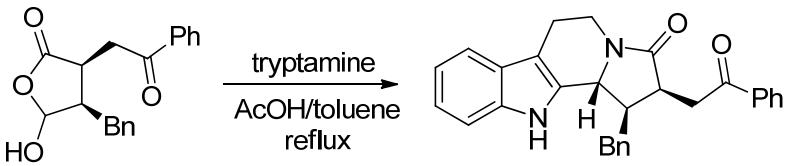



**3a** **6**

To a solution of *tert*-butyl isocyanide (0.24 mmol) in MeOH (2 mL), lactol **3a** (0.2 mmol) was added and the reaction mixture was stirred at 50 °C. Upon the completely consumption of **3a** by TLC, the solvent was evaporated in vacuo and the residue was purified by flash column chromatography giving the 3,4,5-trisubstituted γ-lactone **6** as a colorless oil.

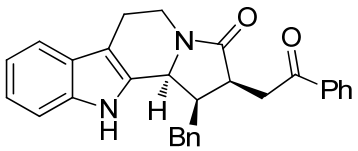



(2R,3R,4S)-3-benzyl-N-(tert-butyl)-5-oxo-4-(2-oxo-2-phenylethyl)tetrahydrofuran-2-carboxamide (**6**). colorless oil; 77% yield; 99% ee; $[\alpha]_D^{20} = 33.1$, (*c* 1.01, $CH_2Cl_2$); $^1H$ NMR (400 MHz, $CDCl_3$): δ 1.33 (s, 9H), 2.55 (dd, *J*= 10.8, 14.0 Hz,1H), 2.77 (dd, *J*= 6.0, 14.0 Hz,1H), 3.26 (dd, *J*= 8.4, 18.4 Hz,1H), 3.43-3.48 (m, 1H), 3.52-3.58 (m, 2H), 4.46 (d, *J*= 2.0 Hz, 1H), 5.88 (s, 1H), 7.16-7.20 ( m, 3H), 7.26-7.30 ( m, 2H), 7.45-7.49 (m, 2H), 7.57-7.61 (m, 1H), 7.93-7.95 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 28.6, 34.6, 34.7, 37.9, 41.6, 51.6, 79.7, 126.8, 128.0, 128.7, 128.7, 128.8, 133.6, 135.8, 137.3, 167.8, 177.4, 196.5 ppm; IR: ν 3404, 2966, 1737, 1686, 1598, 1451, 1372, 1243, 1152, 757, 691 $cm^{-1}$, HRMS calculated for $[C_{24}H_{27}NO_4+H]^+$: 413.1747; found 413.1738. HPLC analysis: Chiralpak AD-H, *n*-hexane/*i*-PrOH=95 / 5, flow rate 1.0 mL/min, λ=254 nm, $t_{major}$= 26.3 min, $t_{minor}$= 35.2 min.




**3a** **7**

To a stirred suspension of **3a** (0.3 mmol) in AcOH/toluene (6 mL, 1:1) was added tryptamine (0.4 mmol). The resultant solution was heated at 80 $^{o}$C for 12h and then the solvent was evaporated in vacuo. The residue was purified by column chromatography to give the product **7** as a pale white solid. The enantioselectivity of the product was determined via the transformation of **7** to its N-Boc derivative.




(1R,2S,11bS)-1-benzyl-2-(2-oxo-2-phenylethyl)-5,6,11,11b-tetrahydro-1H-indolizino[8,7-b]indol-3(2H)-one (**7**). white solid; 45% yield; 98% ee; $[\alpha]_D^{20}$ = 5.76, (*c* 1.04, $CH_2Cl_2$); $^1$H NMR (400 MHz, $CDCl_3$): δ 2.55-2.61 (m, 1H), 2.71 (dd, *J*= 6.4, 15.2 Hz,1H), 2.82-2.97 (m, 2H), 3.02-3.09 (m, 2H), 3.44-3.49 (m, 1H), 3.50-3.61 (m, 2H), 4.56-4.60 (m, 2H), 4.82 (d, *J*= 6.4 Hz, 1H), 5.78 (s, 1H), 6.87 (d, *J*= 4.0 Hz, 1H), 7.01-7.06 ( m, 2H), 7.36-7.38 ( m, 3H), 7.47-7.51 (m, 5H), 7.58-7.59 (m, 1H), 8.01-8.03 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ 20.9, 36.4, 36.9, 38.39, 41.6, 43.0, 59.6, 108.3, 110.6, 118.1, 119.4, 121.8, 126.2, 127.3, 128.0, 128.7, 129.0, 129.7, 132.3, 133.4, 135.4, 136.2, 139.8, 174.4, 198.4 ppm; IR: ν 3424, 3289, 2920, 2245, 1677, 1447, 1302, 1212, 909, 734, 690 cm$^{-1}$, HRMS calculated for $[C_{30}H_{30}N_2O_2+H]^+$: 435.2067; found 435.2076. HPLC analysis: Chiralpak ID, *n*-hexane/$CH_2Cl_2$/MeOH=85/14.5/0.5, flow rate 1.0 mL/min, λ=245 nm, $t_{major}$= 31.2 min, $t_{minor}$= 34.3 min.

**Reference**:

1.Messer, R. Schmitz, A. Moesch, L. Häner, R. *J. Org. Chem.* **2004**, *69*, 8558.

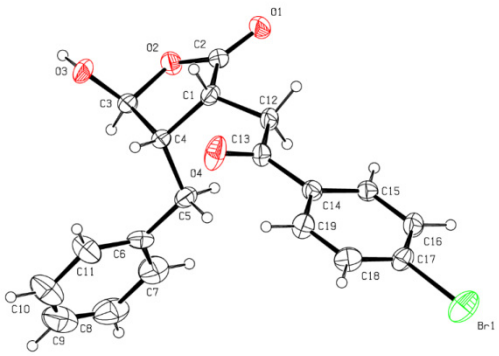
2.(a) Marigo, M., Wabnitz, T. C., Fielenbach, D., Jørgensen, K. A., *Angew. Chem., Int. Ed.*, **2005**, *44*, 794; (b) Hayashi, Y., Gotoh, H., Hayashi, T., Shoji, M., *Angew. Chem., Int. Ed.*, **2005**, *44*, 4212.

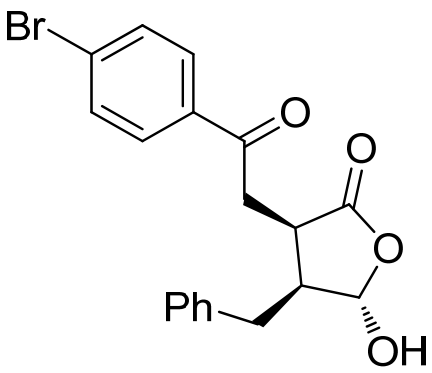
3. Wang, J., Ma, A., Ma, D. *Org. Lett.* **2008**, *10*, 5425.

## 5. Determination of Absolute Configuration of **3a** and **5**.

5.1 The product *ent*-3e was obtained as a white solid using 10 mol% of (*R*)-prolinol silyl ether according to general procedure.

X-ray Crystallographic Data of *ent*-**3e** (CCDC 926785)




| | | | |
|---|---|---|---|
| Bond precision: | C-C = 0.0099 A | | Wavelength=0.71070 |
| Cell: | a=5.8959(3) | b=11.7317(6) | c=12.7300(9) |
| | alpha=90 | beta=96.833(6) | gamma=90 |
| Temperature: 294 K | | | |

| | Calculated | Reported |
|---|---|---|
| Volume | 874.27(9) | 874.27(9) |
| Space group | P 21 | P 1 21 1 |
| Hall group | P 2yb | P 2yb |
| Moiety formula | C19 H17 Br O4 | C19 H17 Br O4 |
| Sum formula | C19 H17 Br O4 | C19 H16 Br O4 |
| Mr | 389.23 | 388.23 |
| Dx,g cm-3 | 1.479 | 1.475 |
| Z | 2 | 2 |
| Mu (mm-1) | 2.370 | 2.370 |
| F000 | 396.0 | 394.0 |
| F000' | 395.61 | |
| h,k,lmax | 7,14,15 | 7,14,15 |
| Nref | 3597[ 1890] | 2479 |
| Tmin,Tmax | 0.473,0.553 | 0.541,1.000 |
| Tmin' | 0.464 | |

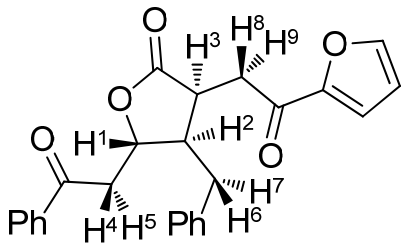
Correction method= MULTI-SCAN

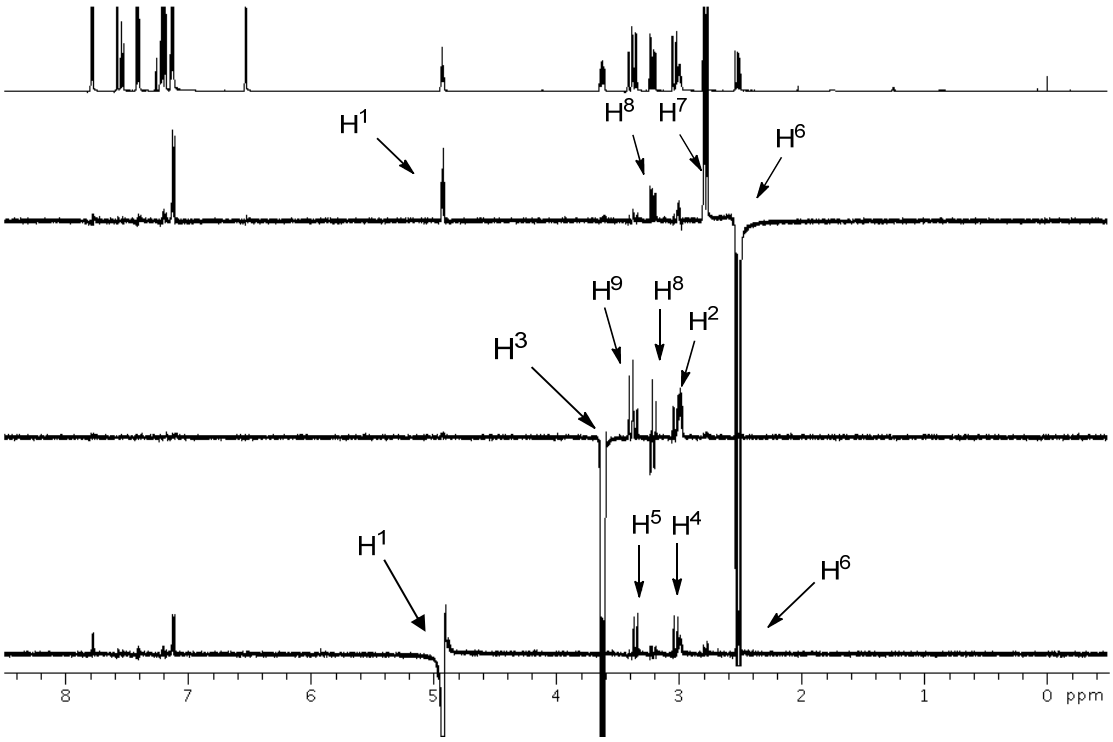
Data completeness= 1.31/0.69 Theta(max)= 26.370

R(reflections)= 0.0522( 1727) wR2(reflections)= 0.1240( 2479)

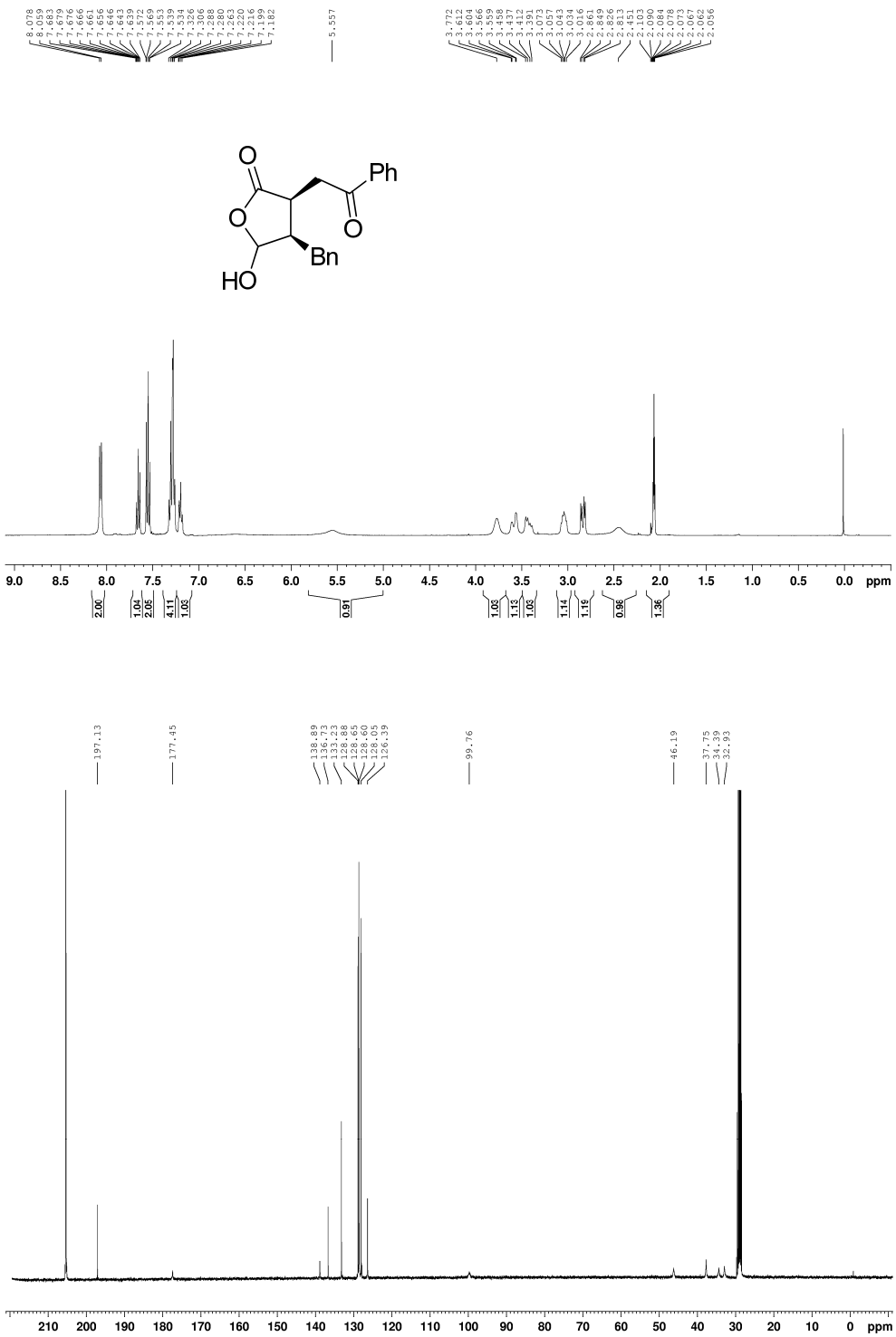
S = 1.084 Npar= Npar = 218

## 5.2 NOE experiments of **5l** (600M Hz, $CDCl_3$)



# 6. NMR Copies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7.683
7.679
7.676
7.666
7.661
7.656
7.646
7.643
7.639
7.572
7.569
7.553
7.539
7.534
7.326
7.306
7.288
7.280
7.263
7.220
7.216
7.199
7.182
5.557
3.772
3.612
3.604
3.566
3.558
3.458
3.437
3.412
3.391
3.073
3.057
3.043
3.034
3.016
2.861
2.849
2.826
2.813
2.451
2.103
2.090
2.084
2.078
2.073
2.067
2.062
2.056
O
O
O
Ph
Bn
HO
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.04
2.06
4.11
1.05
0.91
1.03
1.15
1.05
1.14
1.19
0.98
1.36
197.13
177.45
138.89
136.23
133.23
128.88
128.65
128.60
128.55
126.39
99.76
46.19
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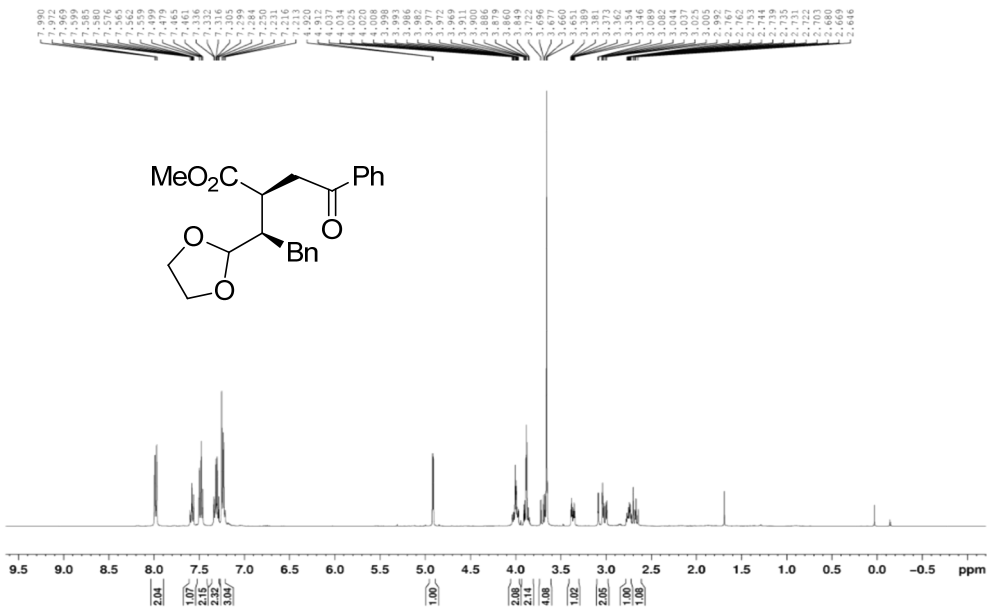
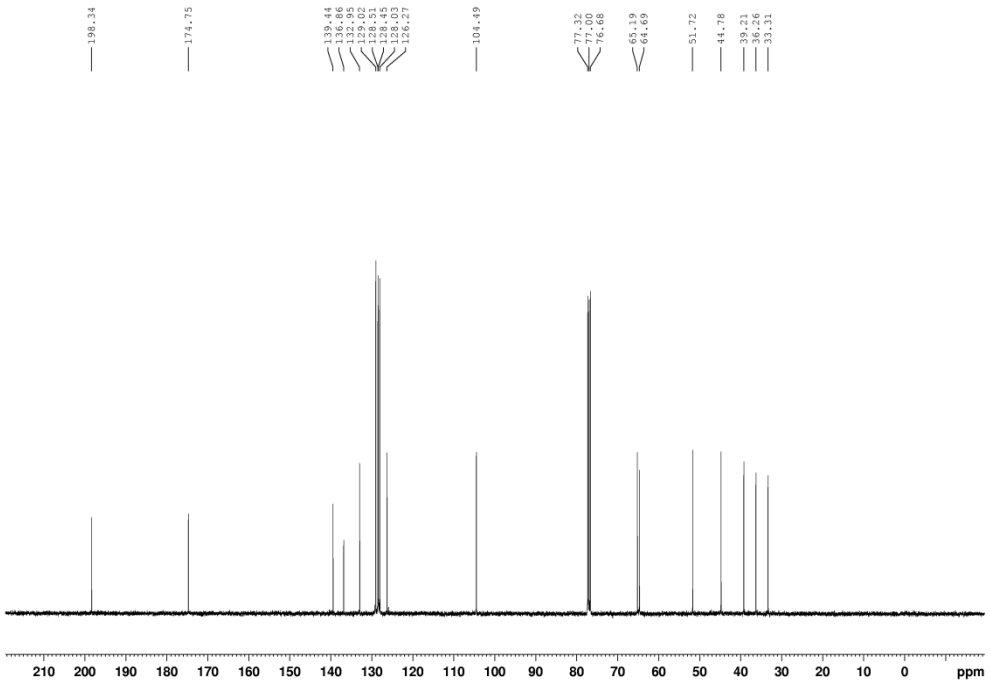
34.39
32.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


MeO2C
Ph
O
O
Bn
O
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm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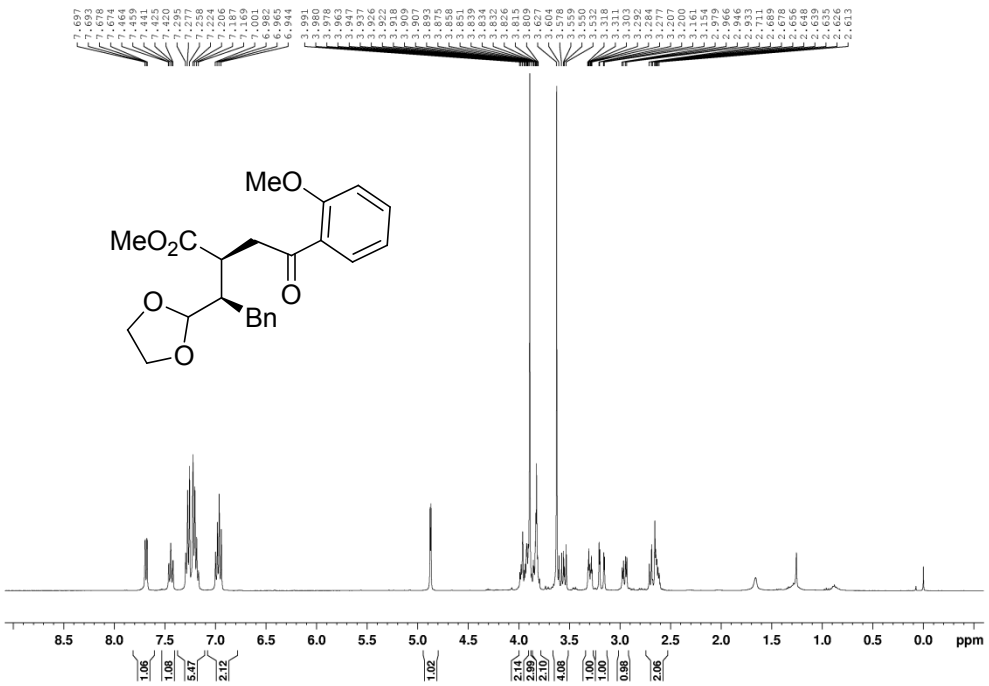
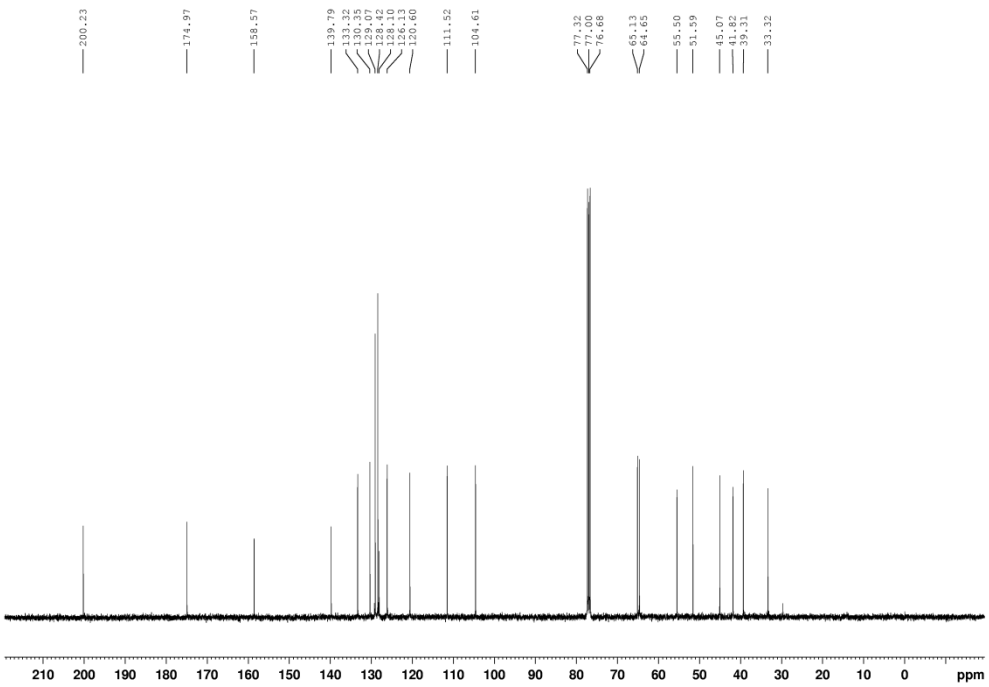
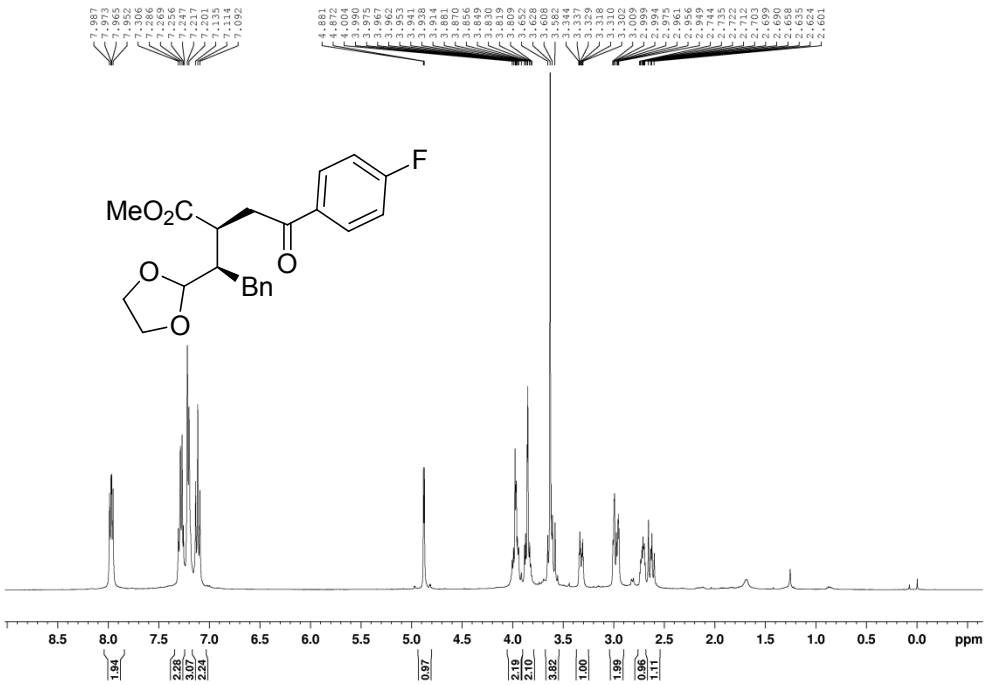
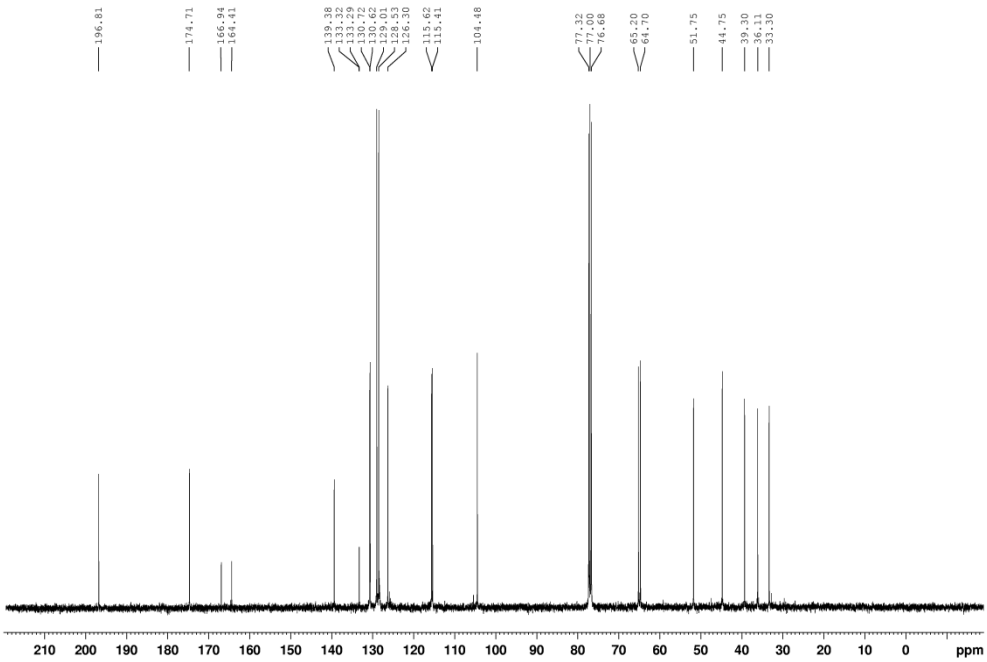
174.75
139.44
136.66
132.95
129.02
128.51
128.45
128.03
126.27
104.49
77.32
77.00
76.68
65.19
64.69
51.72
44.78
39.21
36.26
33.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


MeO
MeO2C
O
O
Bn
O
O




MeO2C
F
O
O
Bn
O
O
ppm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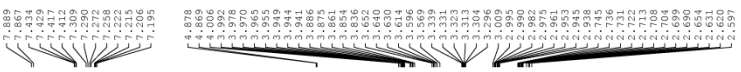
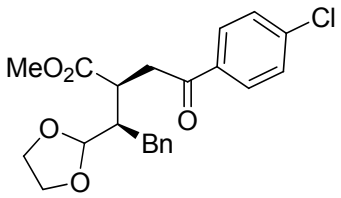
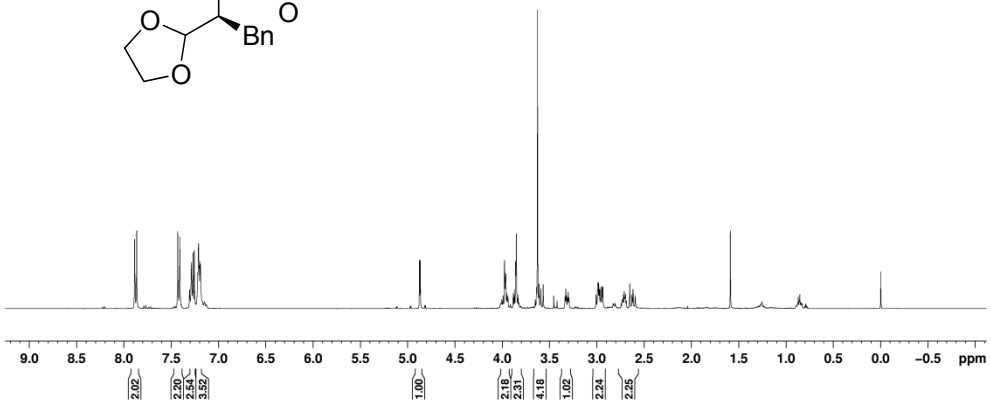
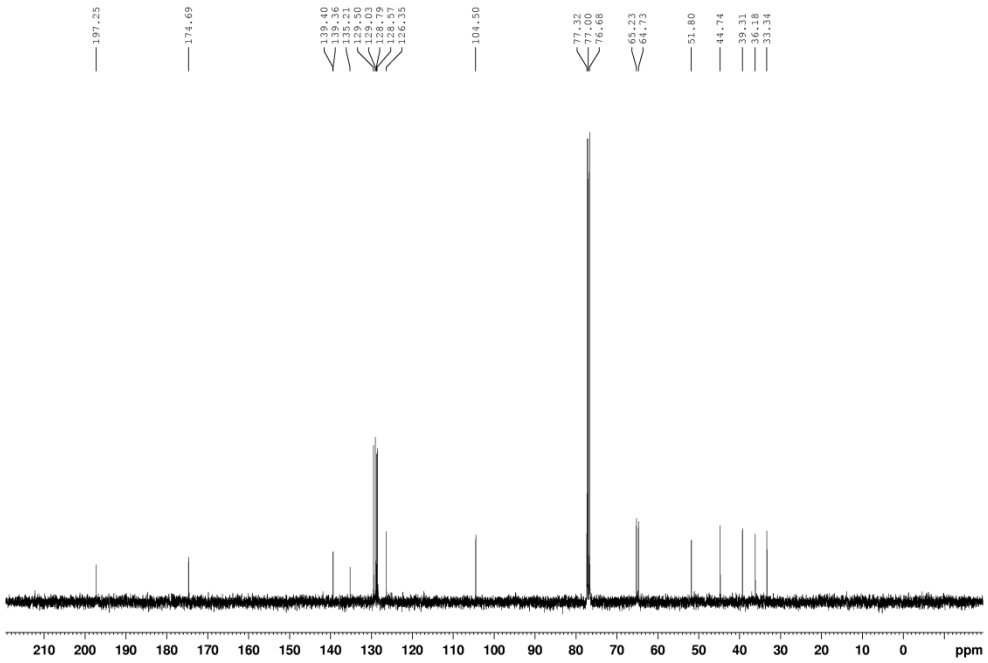
174.71
166.94
164.41
139.38
133.22
133.29
130.72
130.62
129.01
128.53
126.30
115.62
115.41
104.48
77.32
77.00
76.68
65.20
64.70
51.75
44.75
39.30
36.11
33.30
ppm




Cl
MeO2C
O
O
Bn
O
O


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
2.02
2.20
2.54
3.52
1.00
2.18
2.31
4.18
1.02
2.24
2.25


197.25
174.69
139.40
139.36
135.21
129.50
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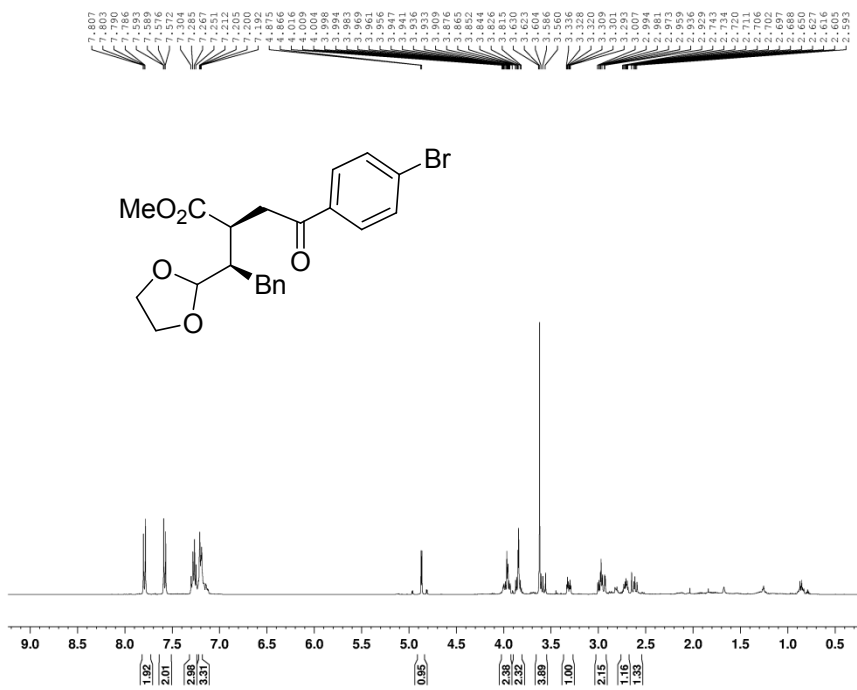
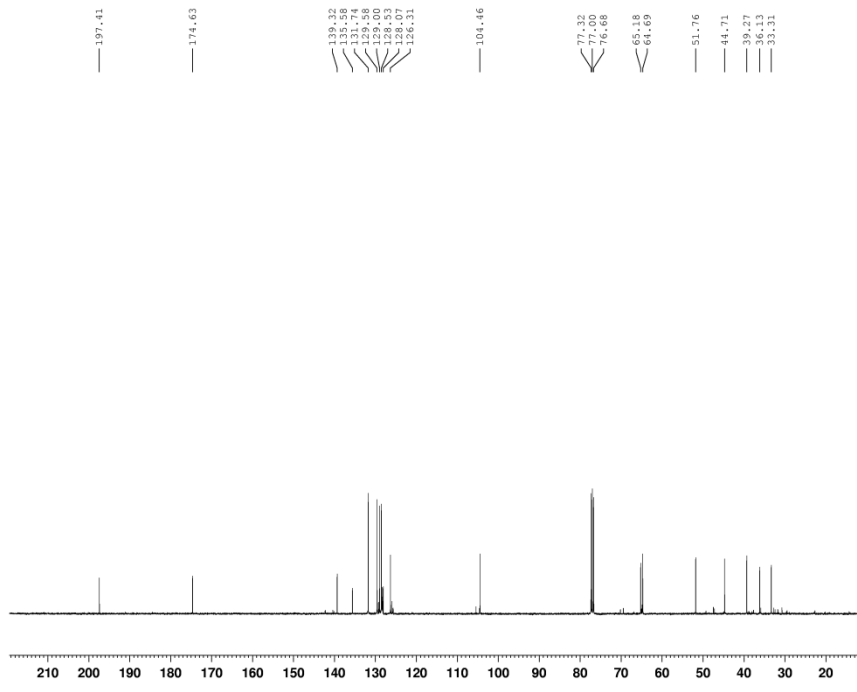
128.79
128.57
126.35
104.50
77.32
77.00
76.68
65.23
64.73
51.80
44.74
39.31
36.18
33.34
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Br
MeO2C
O
O
Bn
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.92 2.01 2.98 3.57 0.95 2.38 2.32 3.89 1.00 2.15 1.16 1.53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174.63
139.32
135.88
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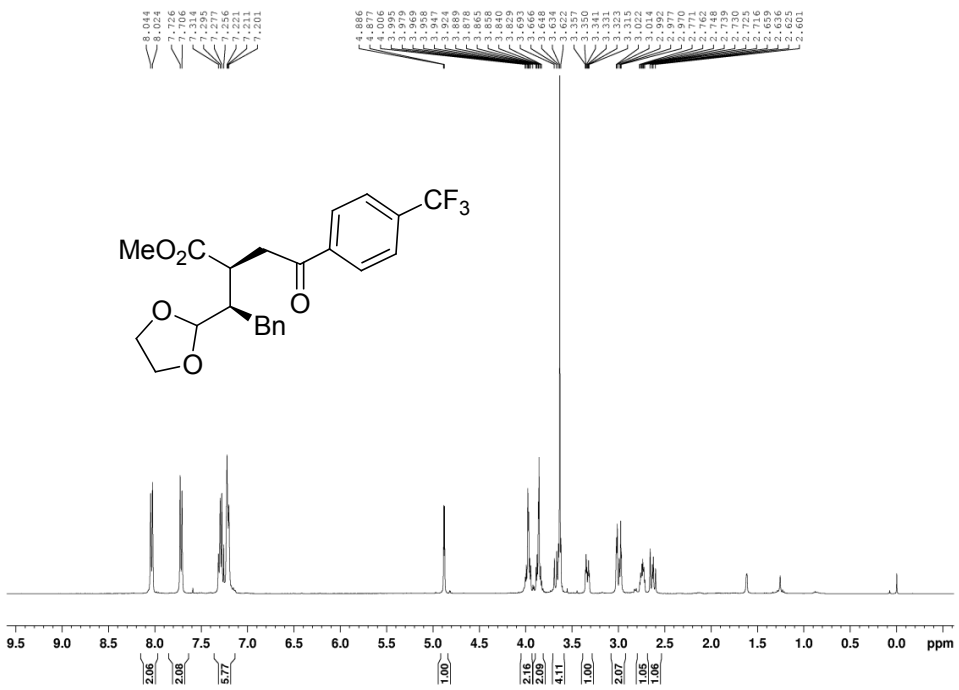
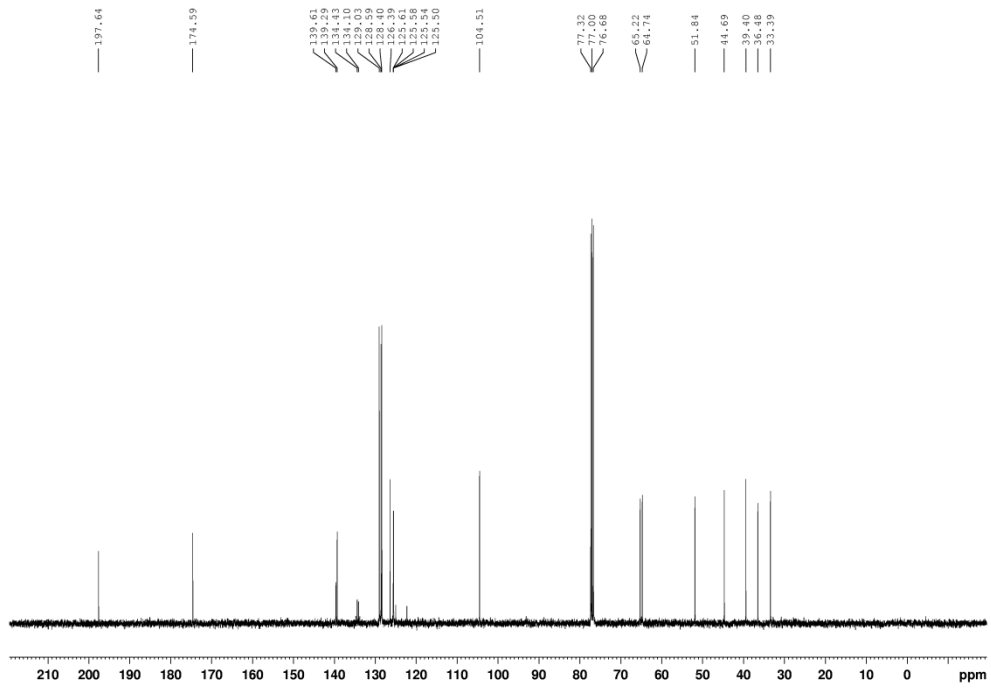
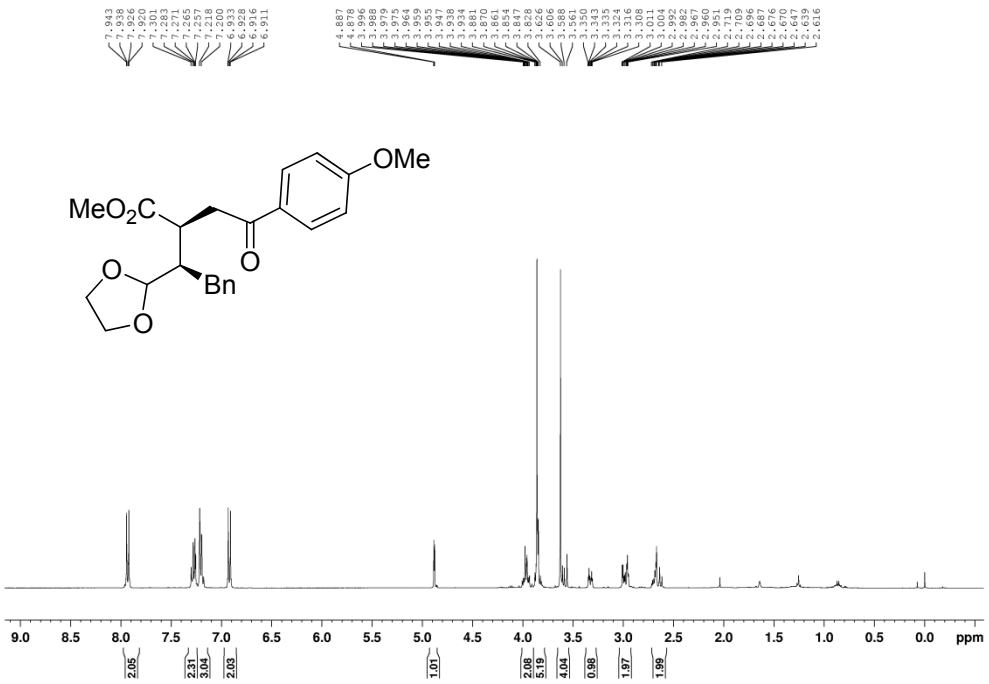
129.58
129.00
128.53
128.07
126.31
104.46
77.32
77.00
76.68
65.18
64.69
51.76
44.71
39.27
36.13
33.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


MeO2C
CF3
O
O
O
O
Bn
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.06 2.08 5.77 1.00 2.16 2.09 4.11 1.00 2.07 1.05 1.06


197.64
174.59
139.61
139.29
134.43
134.10
129.03
128.59
128.40
126.39
125.61
125.58
125.54
125.50
104.51
77.32
77.00
76.68
65.22
64.74
51.84
44.69
39.40
36.48
33.39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.271
7.265
7.257
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7.200
6.933
6.928
6.916
6.911
4.887
4.878
3.996
3.988
3.979
3.975
3.964
3.959
3.955
3.947
3.938
3.934
3.881
3.870
3.861
3.854
3.847
3.828
3.626
3.606
3.588
3.561
3.350
3.343
3.335
3.324
3.316
3.308
3.011
3.004
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2.967
2.960
2.951
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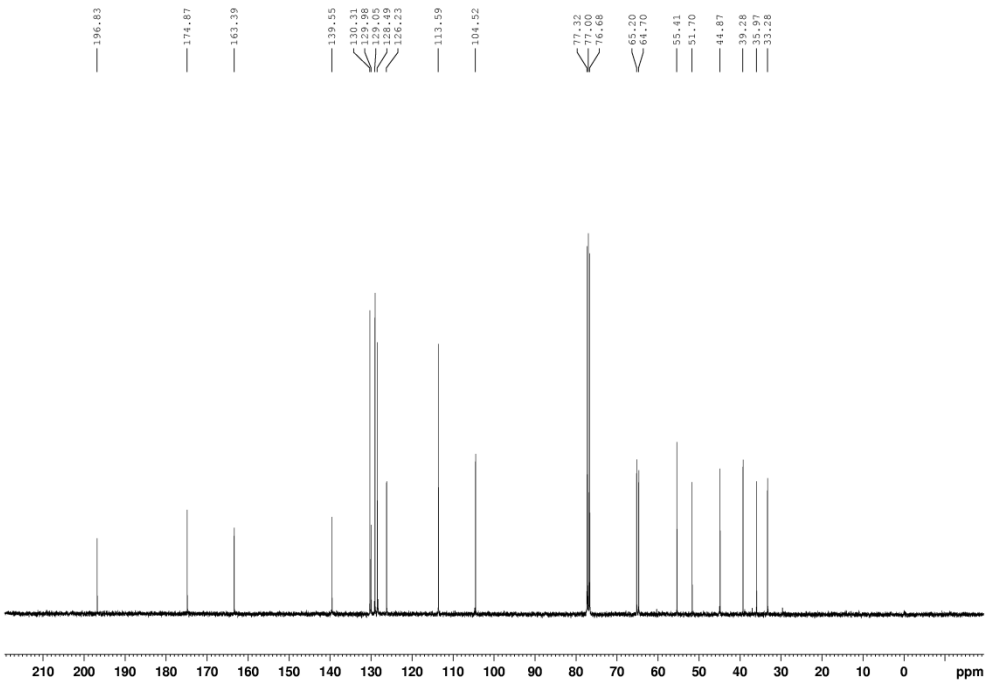
2.696
2.687
2.676
2.670
2.647
2.639
2.616
OMe
MeO2C
O
O
Bn
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.05
2.31
3.04
2.03
1.01
2.08
5.19
4.04
0.98
1.97
1.99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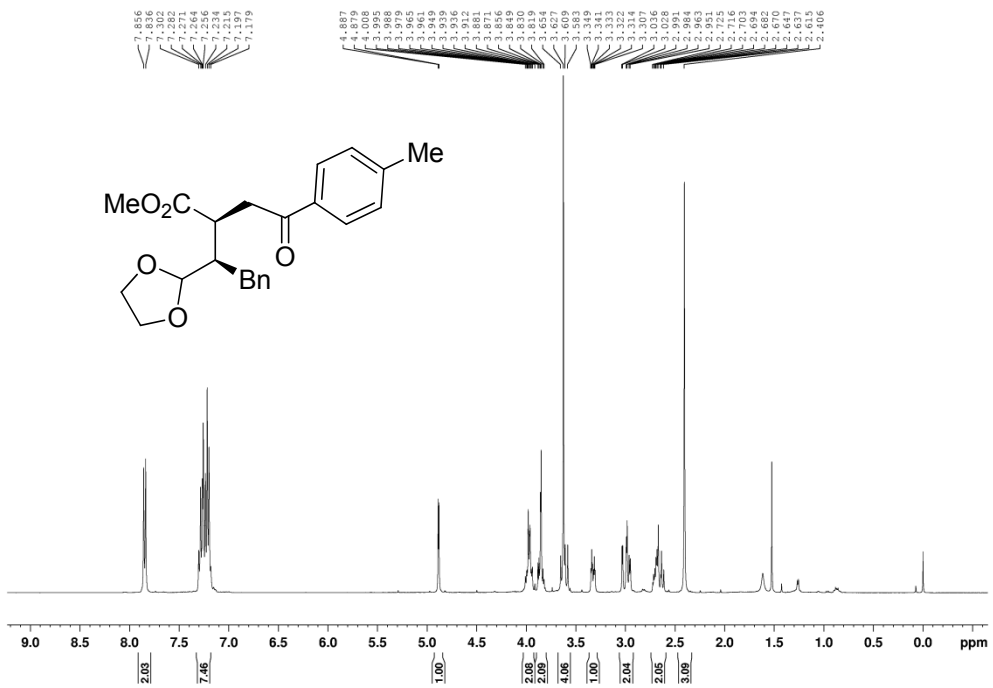
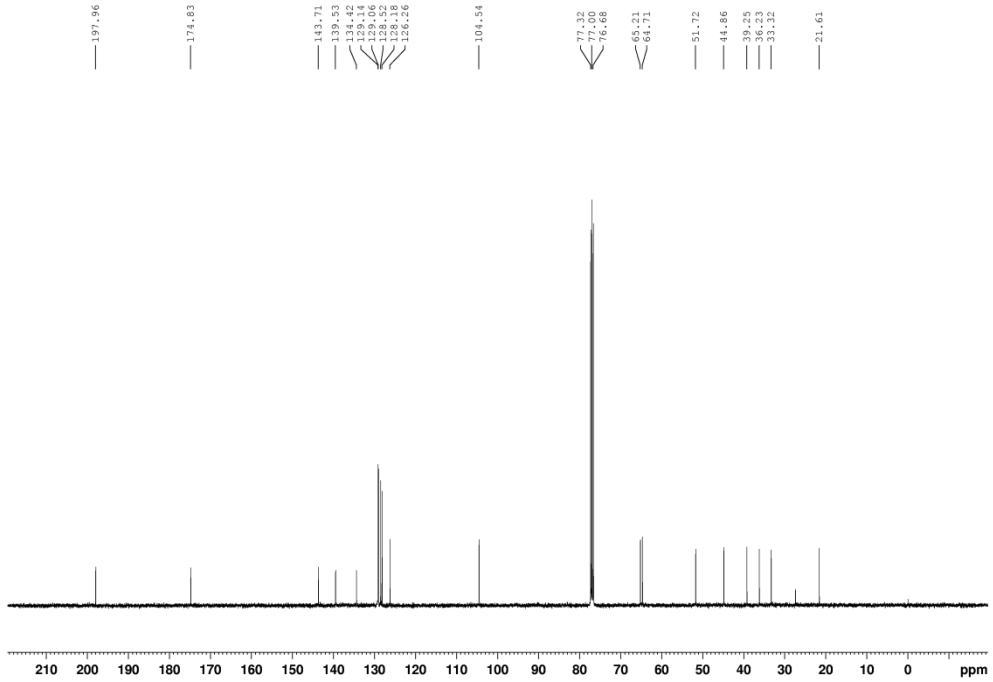
128.49
126.23
113.59
104.52
77.32
77.00
76.68
65.20
64.70
55.41
51.70
44.87
39.28
35.97
33.28
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


Me
MeO2C
O
Bn
O
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.03 7.46 1.00 2.08 2.09 4.06 1.00 2.04 2.05 3.09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139.53
134.42
129.14
129.06
128.52
128.18
126.26
104.54
77.32
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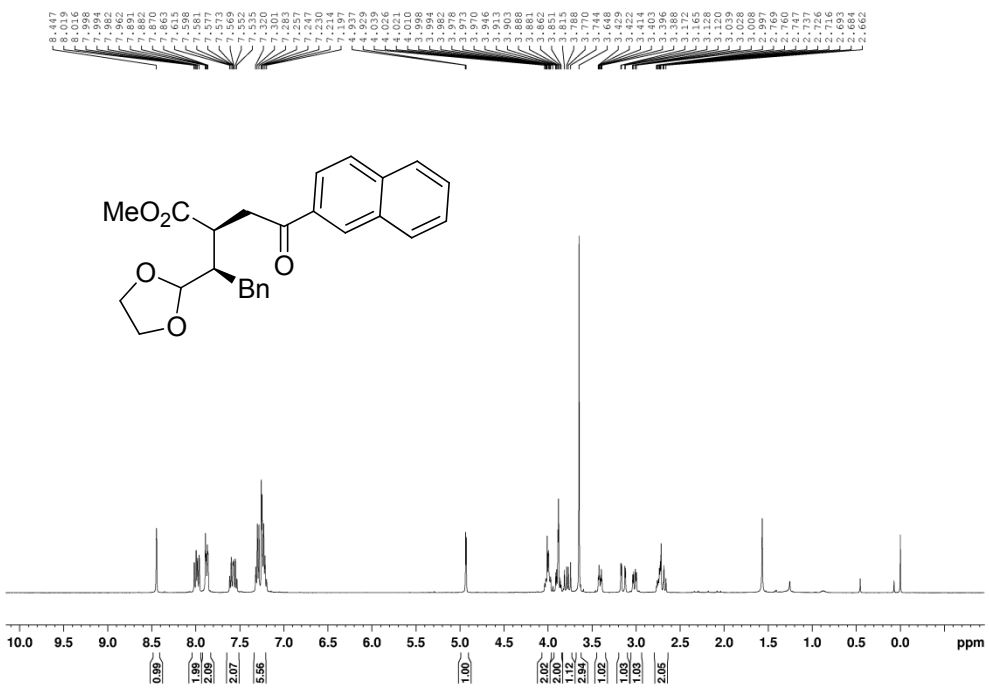
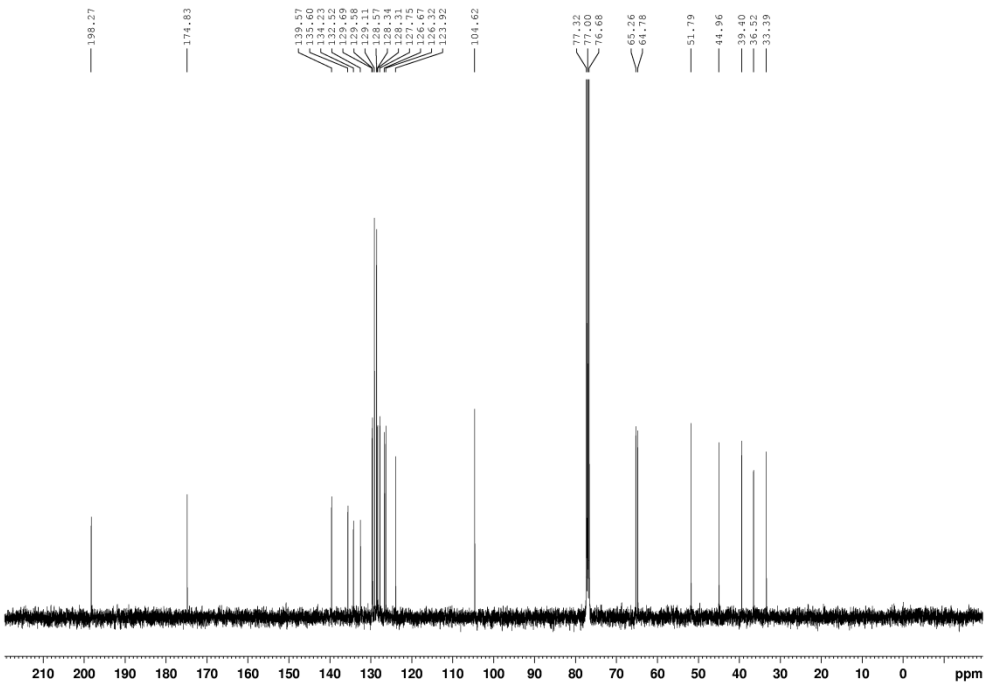
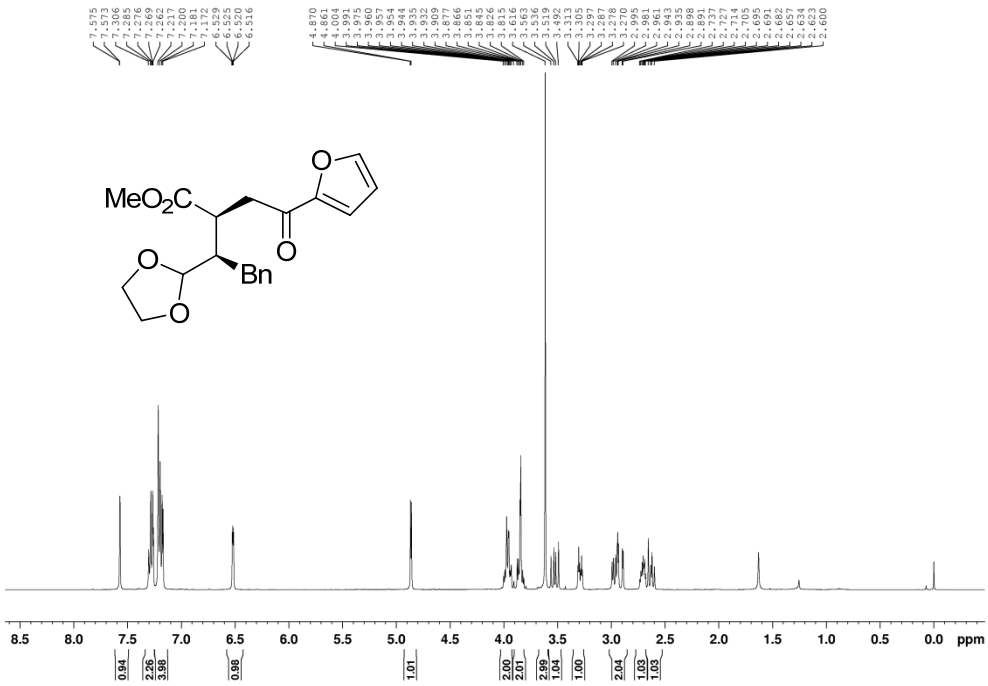
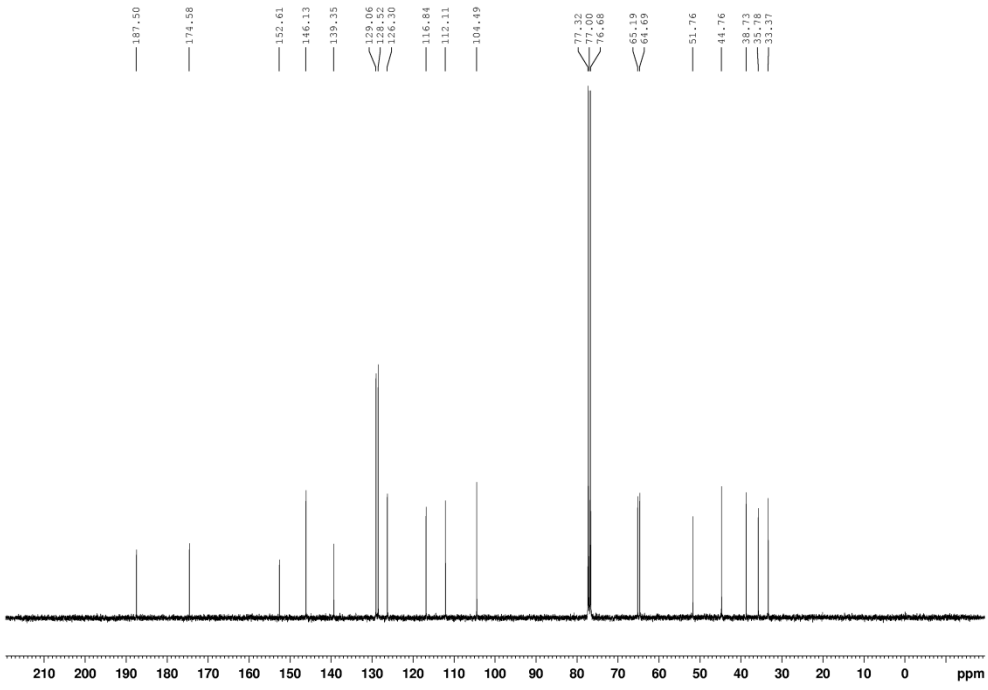
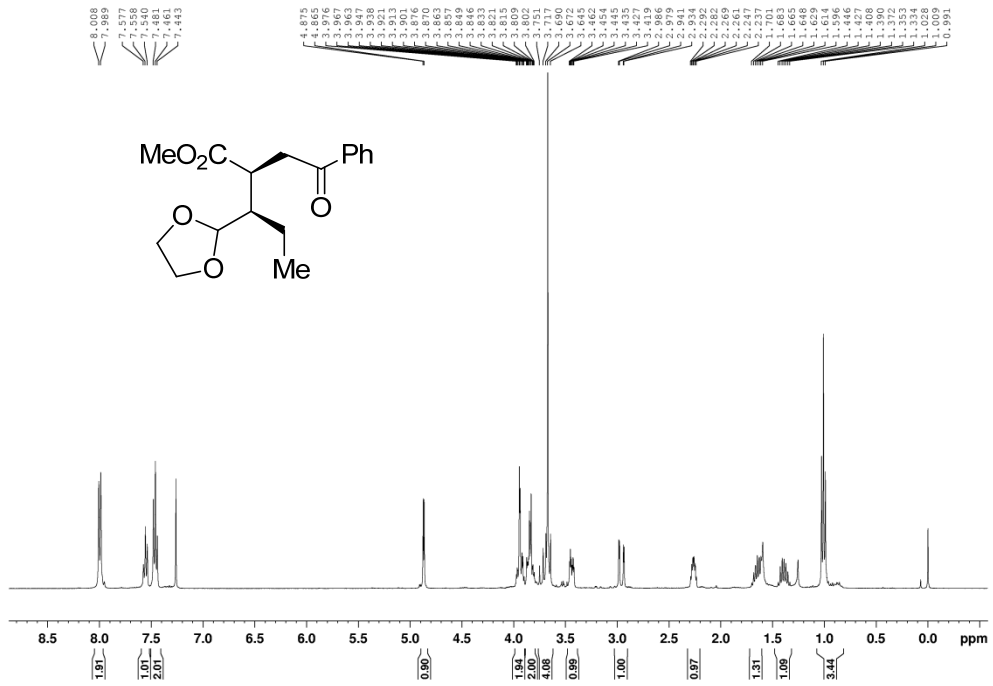
76.68
65.21
64.71
51.72
44.86
39.25
36.23
33.32
21.61
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

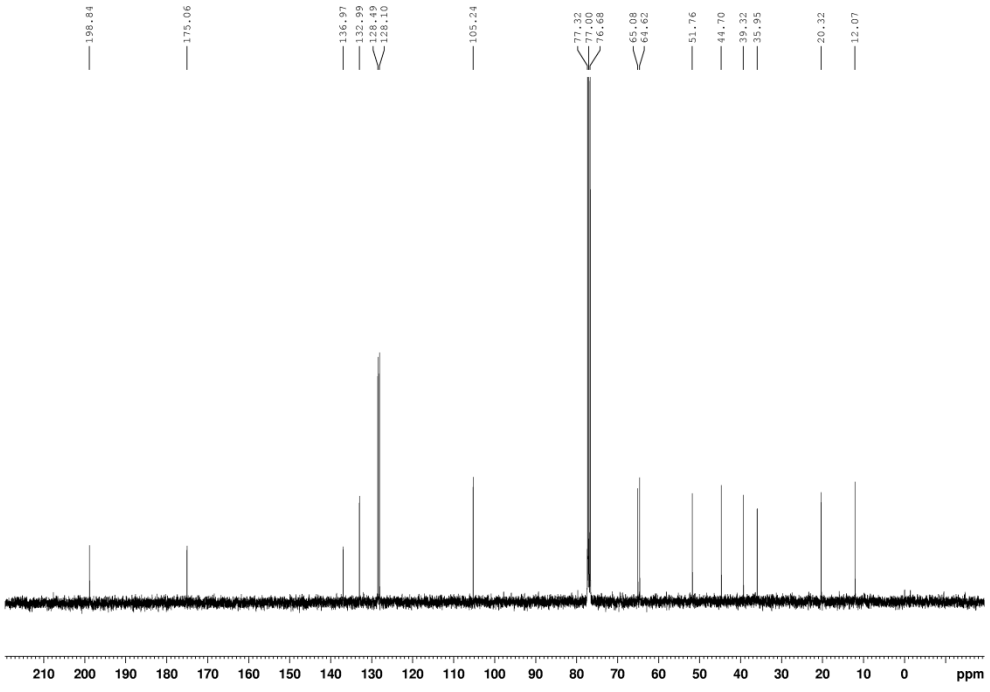

MeO2C
O
O
Bn
O
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

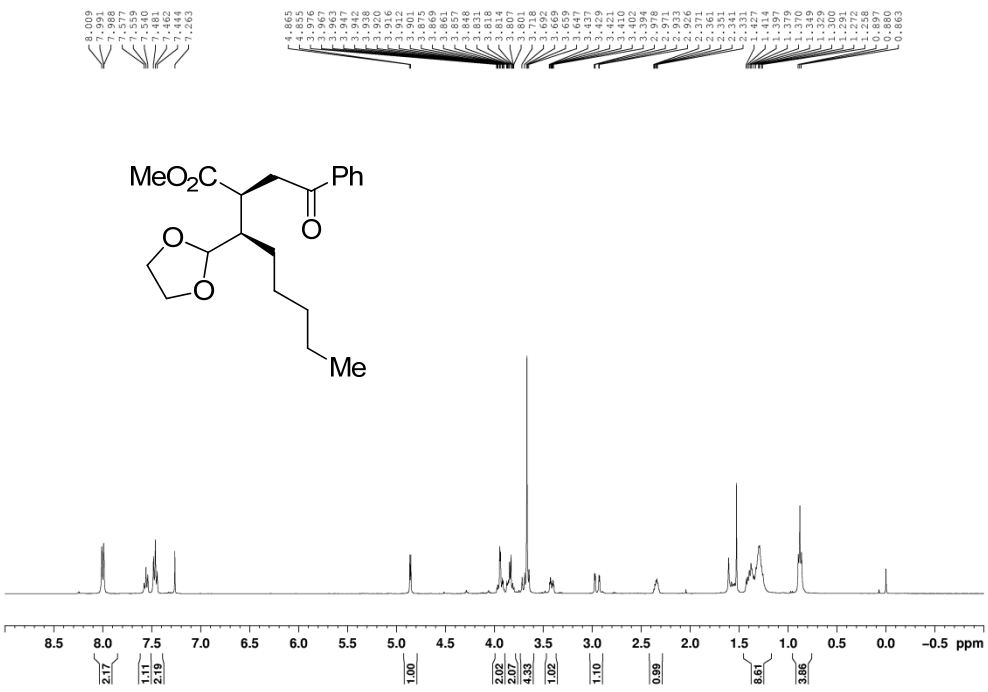

198.27
174.83
139.57
135.60
134.23
132.52
129.69
129.38
129.31
128.57
128.34
128.31
127.75
126.67
126.32
123.92
104.62
77.32
77.00
76.68
65.26
64.78
51.79
44.96
39.40
36.52
33.39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

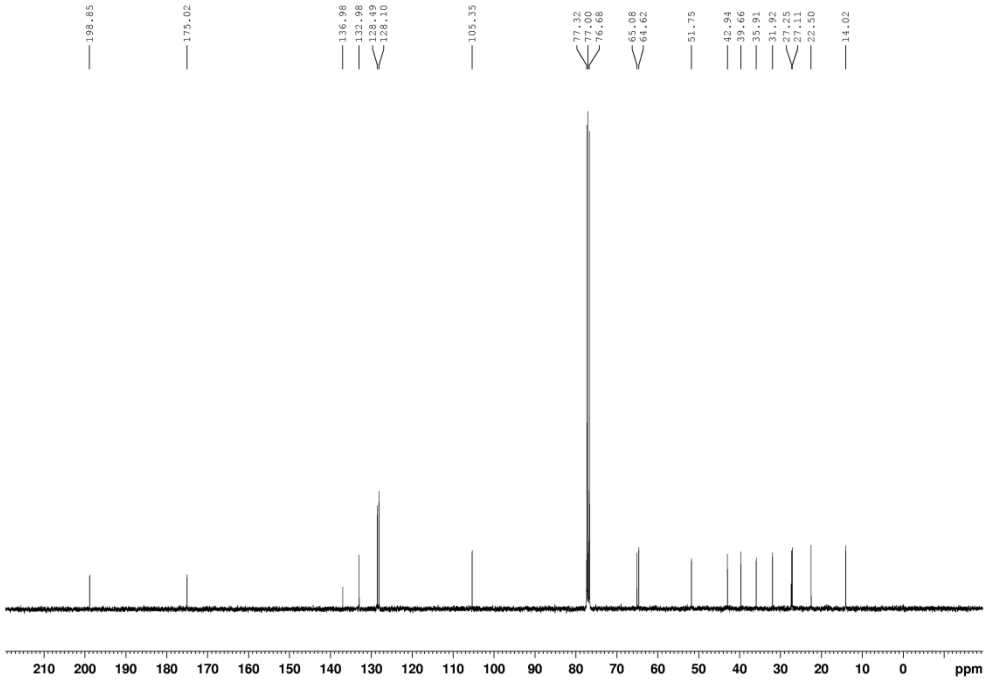

MeO2C
O
O
O
Bn
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

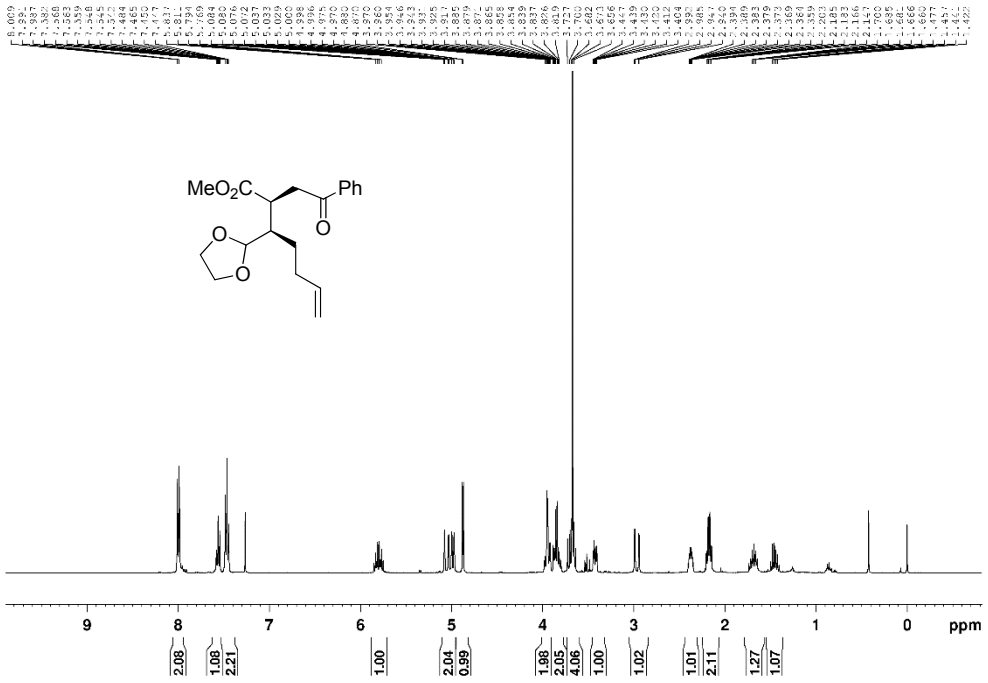

187.50
174.58
152.61
146.13
139.35
129.06
128.52
126.30
116.84
112.11
104.49
77.32
77.00
76.68
65.19
64.69
51.76
44.76
38.73
35.78
33.37
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

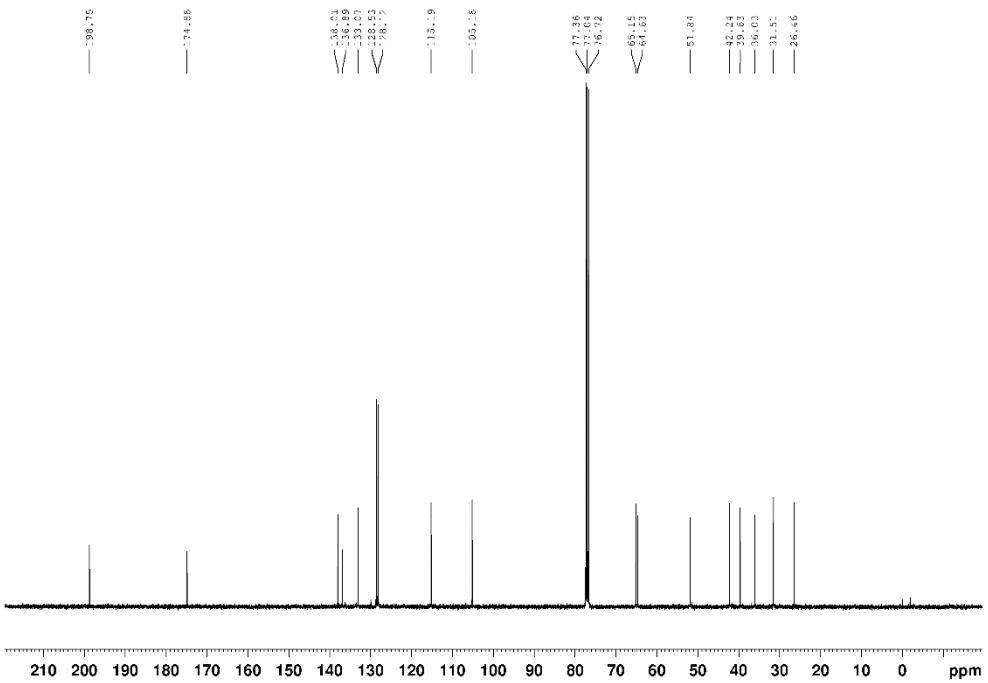

MeO2C
Ph
O
O
O
Me
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

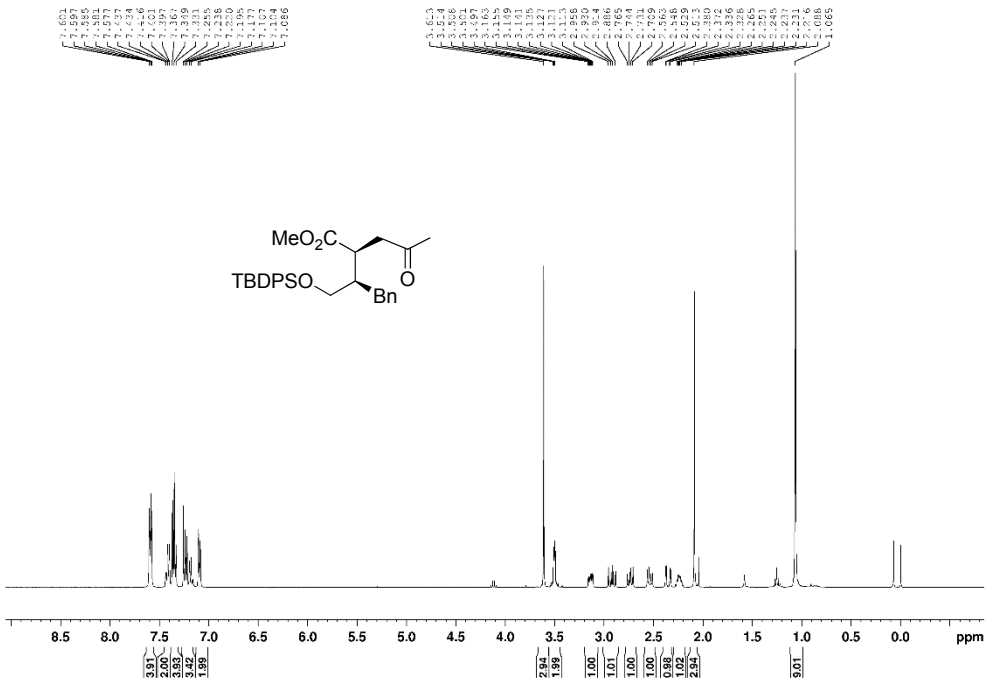

198.84
175.06
136.97
132.99
128.49
128.10
105.24
77.32
77.00
76.68
65.08
64.62
51.76
44.70
39.32
35.95
20.32
12.07
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

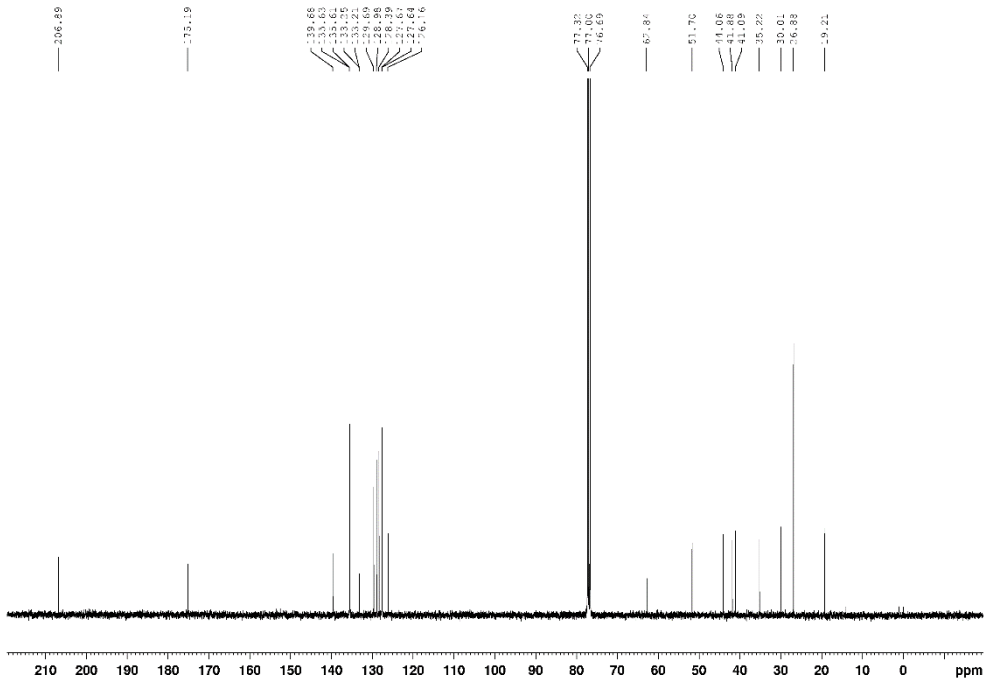

MeO2C
Ph
O
O
O
Me
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm

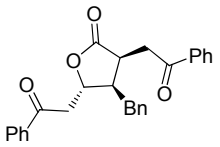

198.85
175.02
136.98
132.98
128.49
128.10
105.35
77.32
77.00
76.68
65.08
64.62
51.75
42.94
39.66
35.91
31.92
27.25
27.11
22.50
14.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

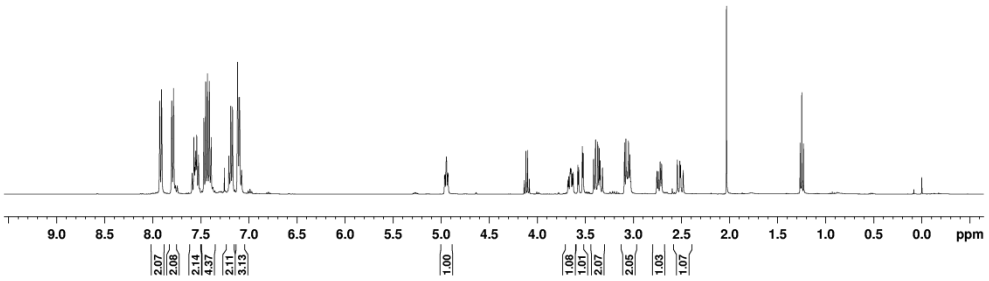

MeO2C
Ph
O
O
O
9
8
7
6
5
4
3
2
1
0
ppm
2.08
1.08
2.21
1.00
2.04
0.99
1.98
2.05
4.06
1.00
1.02
1.01
2.11
1.27
1.07

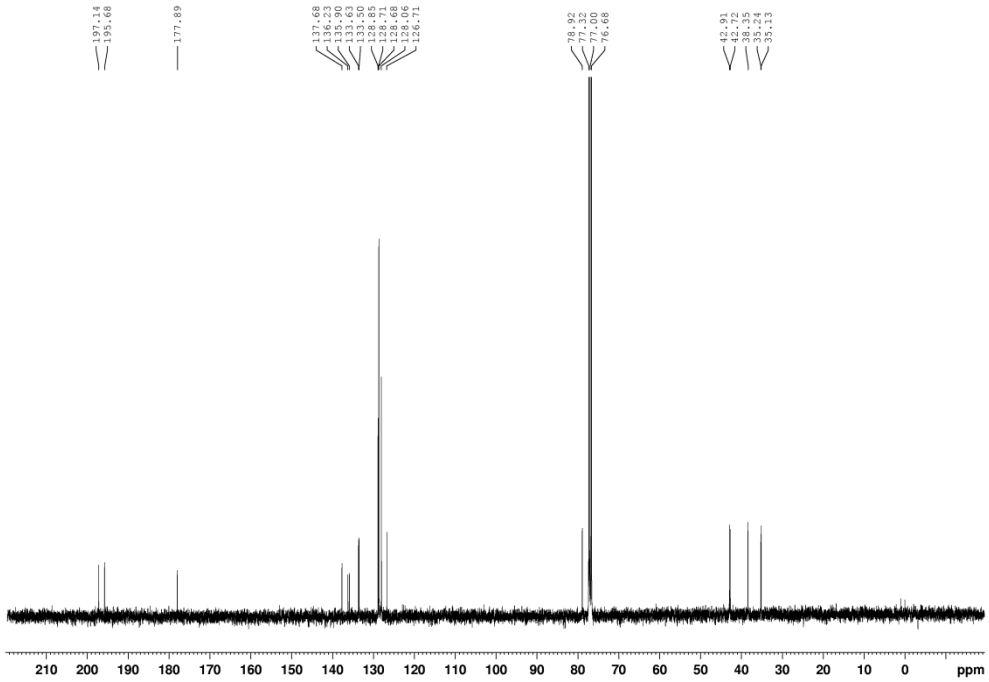

198.75
174.88
138.01
136.89
129.07
128.53
128.17
115.19
105.18
77.36
77.04
76.72
65.15
61.63
51.84
42.24
39.63
36.03
31.51
26.46
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

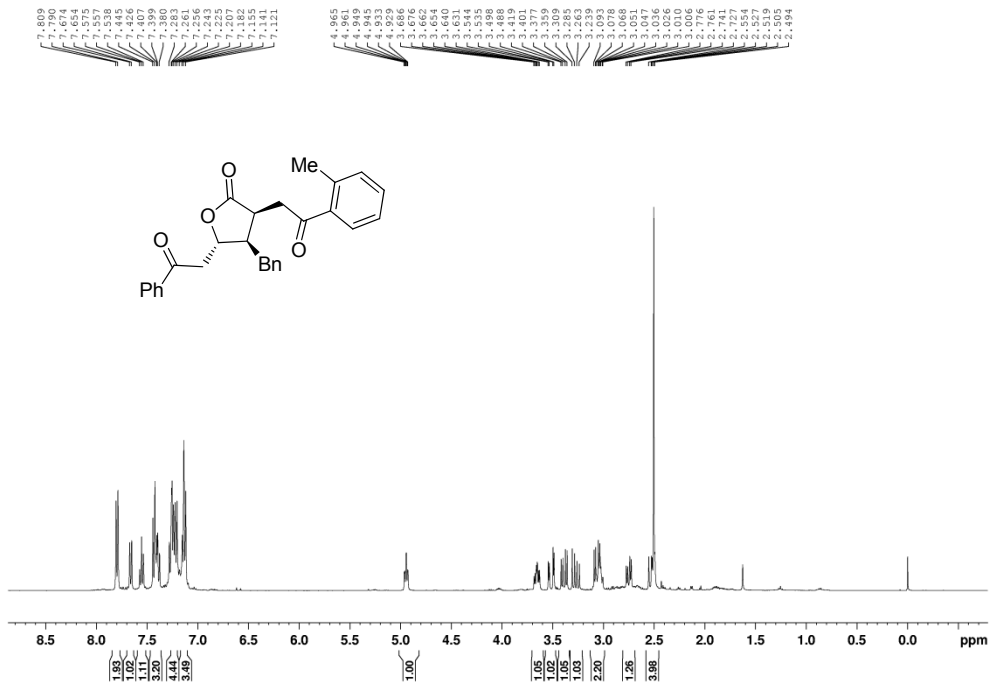

MeO2C
TBDPSO
Bn
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

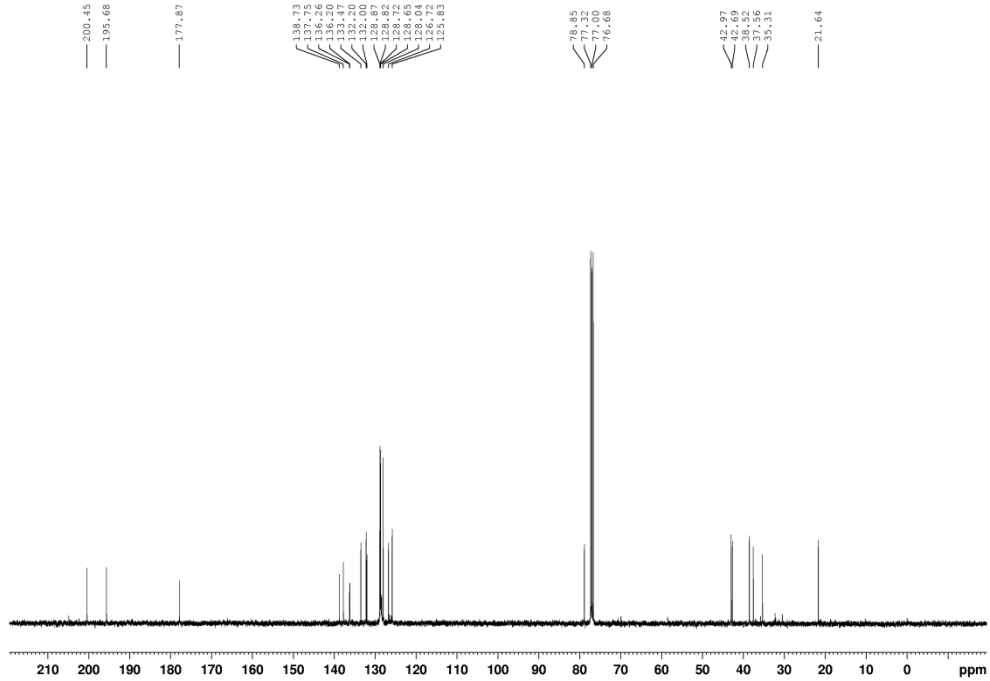

206.89
175.19
77.32
77.00
76.69
67.84
51.70
41.06
41.88
41.09
35.22
30.01
26.88
19.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

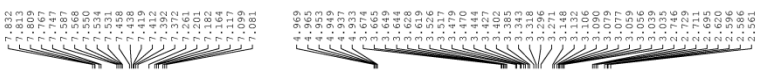

O
O
Ph
O
O
Bn
Ph


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.07 2.08 2.14 4.37 2.11 3.19 1.00 1.08 1.01 2.07 2.05 1.03 1.07

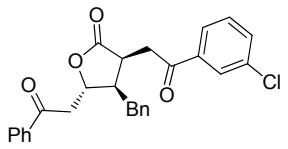

197.14
195.68
177.89
137.68
136.33
136.09
133.63
133.50
128.85
128.71
128.48
128.06
126.71
78.92
77.32
77.00
76.68
42.91
42.72
38.35
35.24
35.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

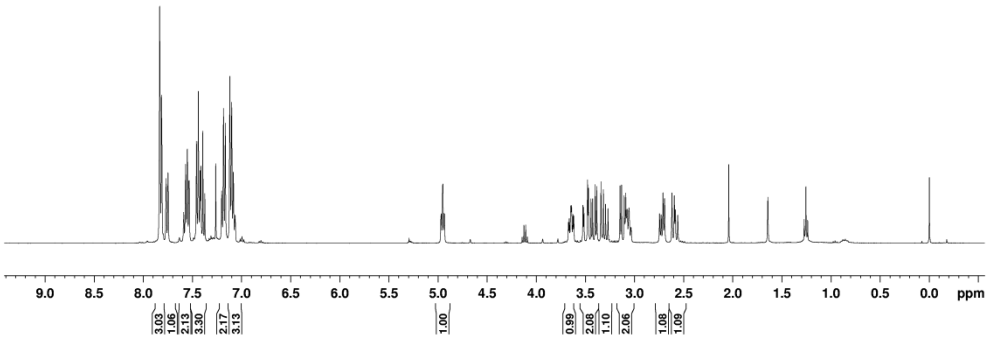

Me
O
O
O
O
Bn
Ph
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

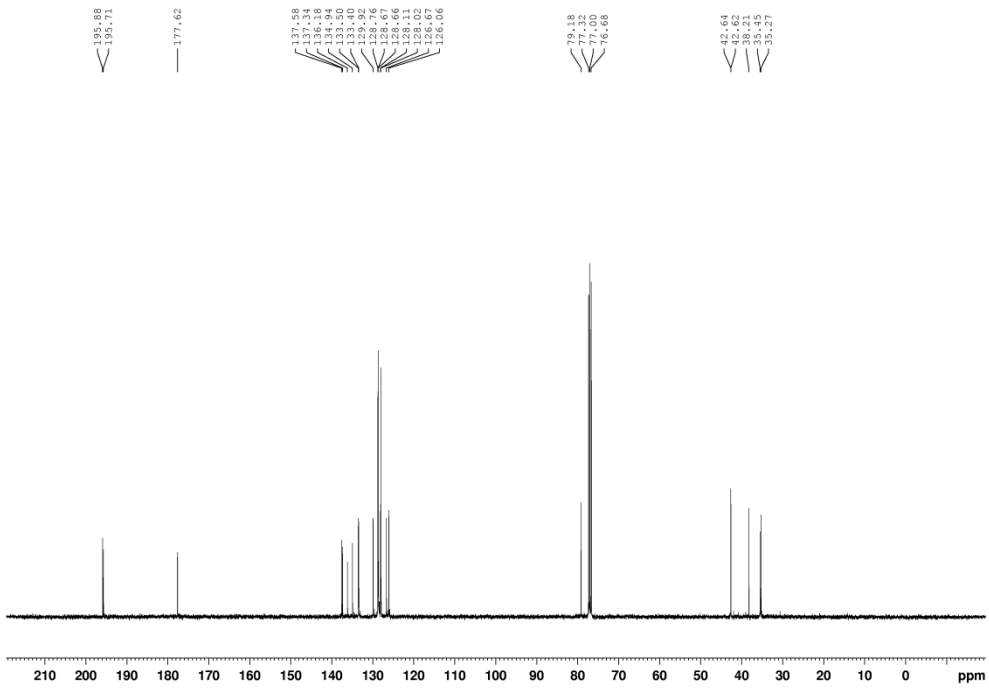

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

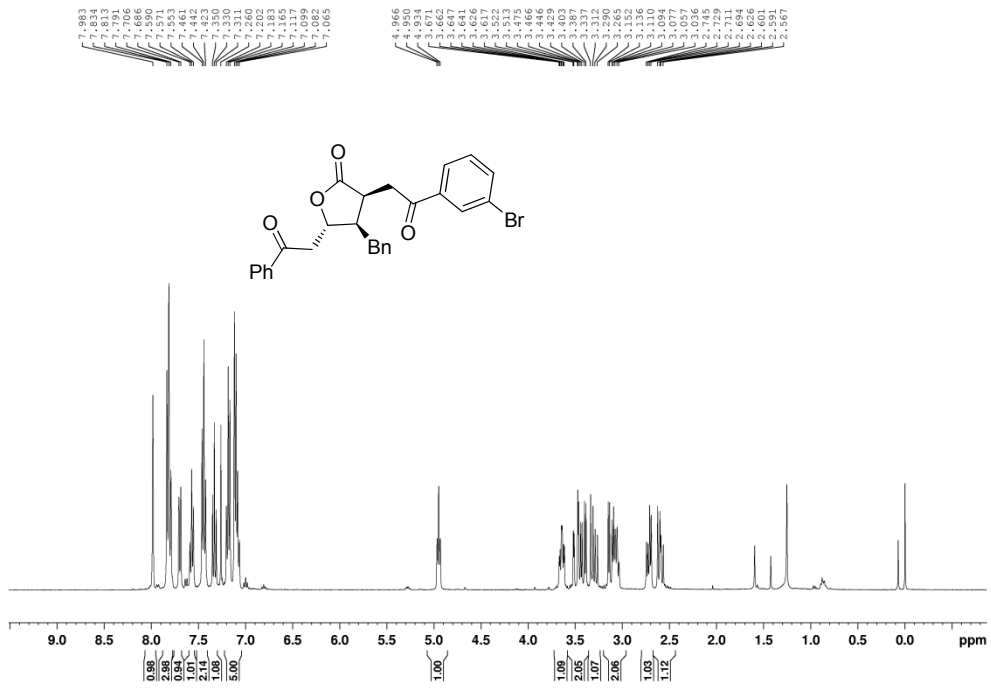


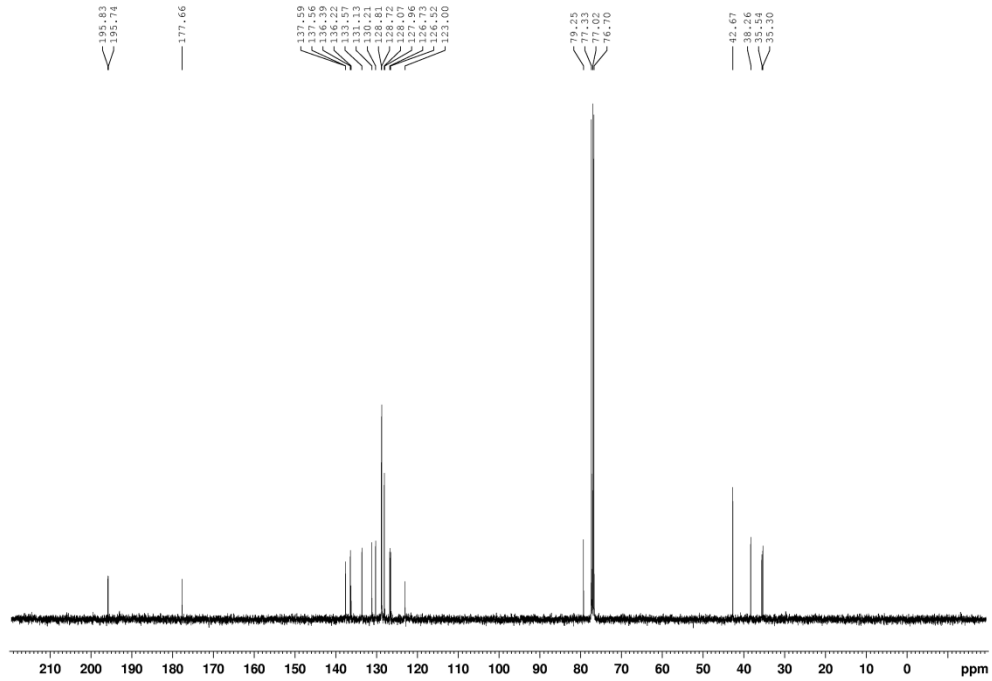


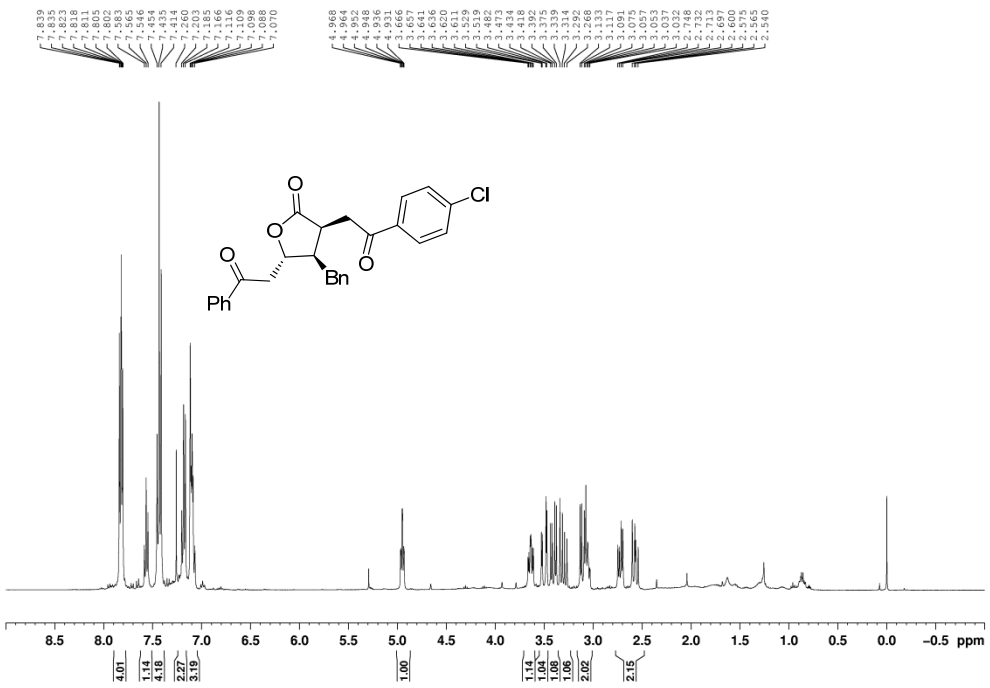

O
O
O
O
Cl
Bn
Ph

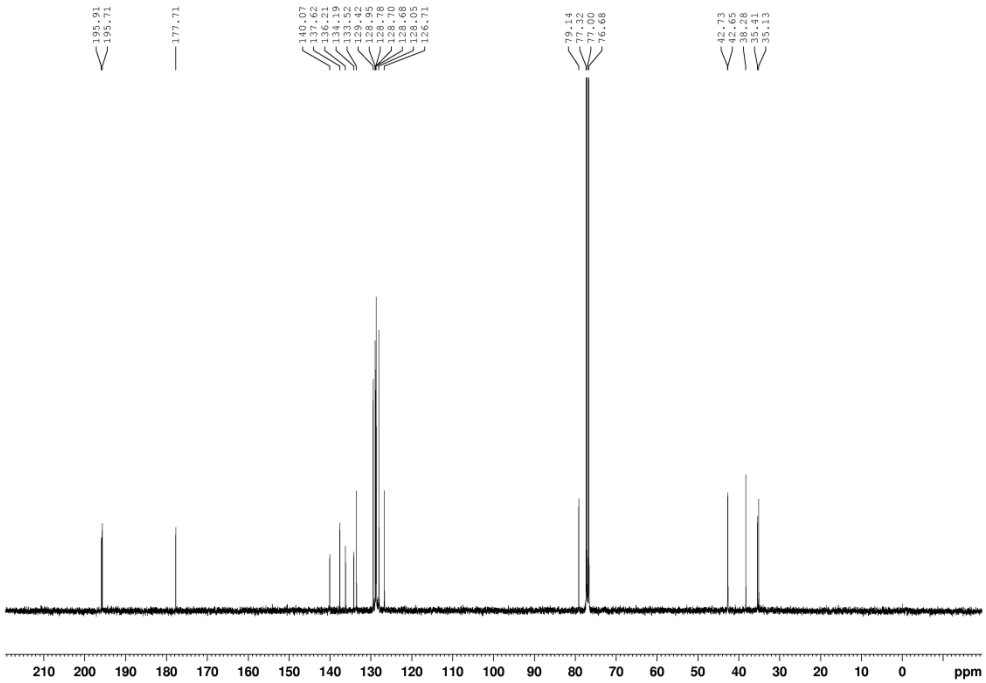

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.03 1.06 2.13 3.30 2.17 3.13 1.00 0.99 2.08 1.10 2.06 1.08 1.09


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

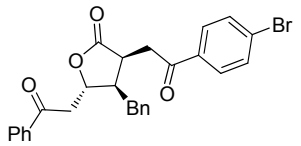

7.983
7.834
7.813
7.791
7.706
7.686
7.590
7.571
7.553
7.461
7.442
7.423
7.350
7.330
7.311
7.260
7.202
7.183
7.165
7.117
7.099
7.080
7.065
4.966
4.950
4.934
3.671
3.662
3.647
3.641
3.626
3.617
3.522
3.513
3.475
3.466
3.446
3.429
3.403
3.387
3.337
3.312
3.290
3.265
3.152
3.136
3.110
3.094
3.077
3.057
3.036
3.015
2.729
2.711
2.694
2.676
2.661
2.591
2.567
O
O
O
O
Br
Bn
Ph
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.98
2.98
0.94
1.01
2.14
1.08
5.00
1.00
1.09
2.05
1.07
2.06
1.03
1.12

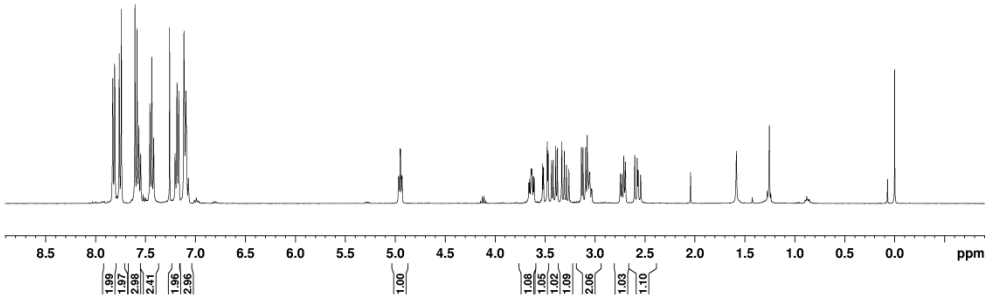

195.83
195.74
177.66
137.59
137.56
136.39
136.22
133.57
133.13
130.21
128.81
128.72
128.07
127.96
126.73
126.52
123.00
79.25
77.33
77.02
76.70
42.67
38.26
35.54
35.30
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

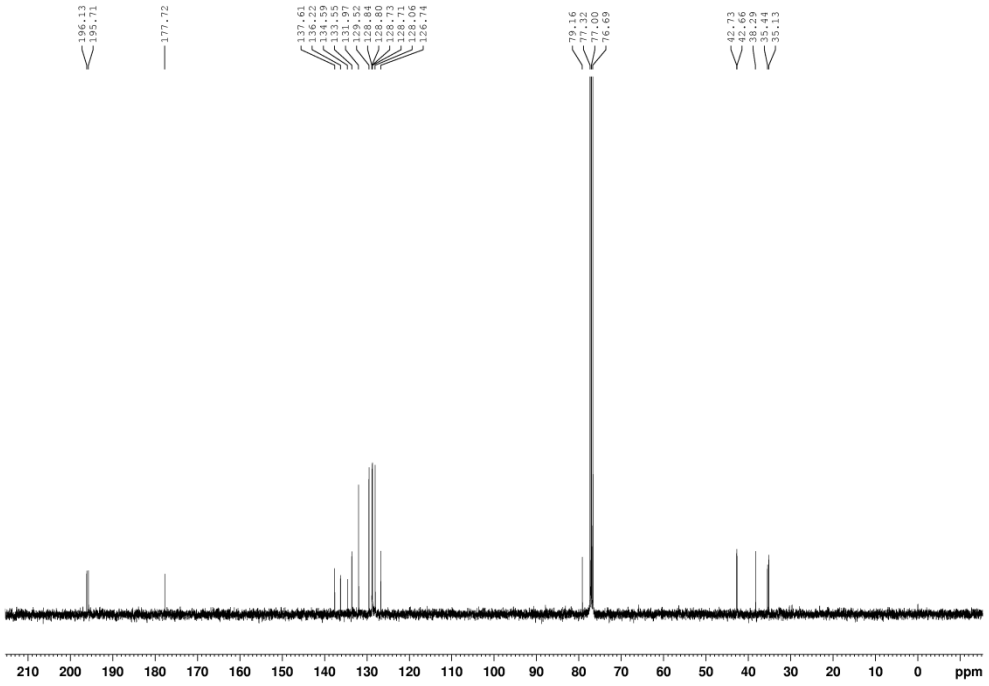

7.839
7.835
7.823
7.818
7.811
7.805
7.802
7.583
7.565
7.546
7.454
7.435
7.414
7.260
7.203
7.185
7.166
7.116
7.109
7.098
7.088
7.070
4.968
4.964
4.952
4.948
4.936
4.931
3.666
3.657
3.641
3.636
3.620
3.611
3.579
3.519
3.482
3.473
3.434
3.418
3.392
3.375
3.339
3.314
3.282
3.268
3.133
3.117
3.091
3.075
3.057
3.053
3.037
3.022
2.748
2.732
2.713
2.697
2.660
2.575
2.565
2.540
O
O
O
O
Cl
Bn
Ph
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
4.01
1.14
4.18
2.27
3.19
1.00
1.14
1.04
1.08
1.06
2.02
2.15

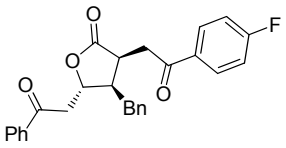

195.91
195.71
177.71
140.07
137.62
136.21
134.19
133.52
129.42
128.95
128.78
128.70
128.68
128.05
126.71
79.14
77.32
77.00
76.68
42.73
40.65
38.28
35.41
35.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

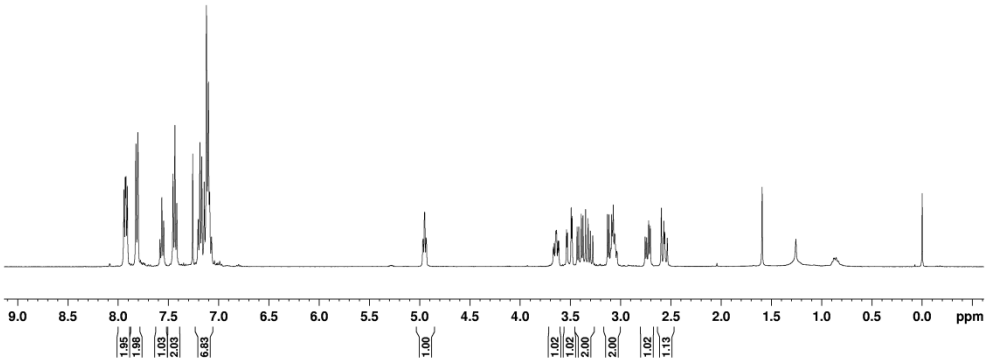

7.827
7.809
7.763
7.741
7.607
7.586
7.569
7.551
7.458
7.439
7.420
7.261
7.206
7.187
7.169
7.117
7.112
7.097
7.092
4.969
4.965
4.953
4.949
4.937
4.932
3.664
3.656
3.640
3.634
3.619
3.610
3.526
3.517
3.479
3.470
3.436
3.419
3.394
3.377
3.333
3.309
3.287
3.262
3.136
3.121
3.094
3.078
3.057
3.053
3.037
3.032
2.747
2.731
2.712
2.697
2.693
2.578
2.568
2.543

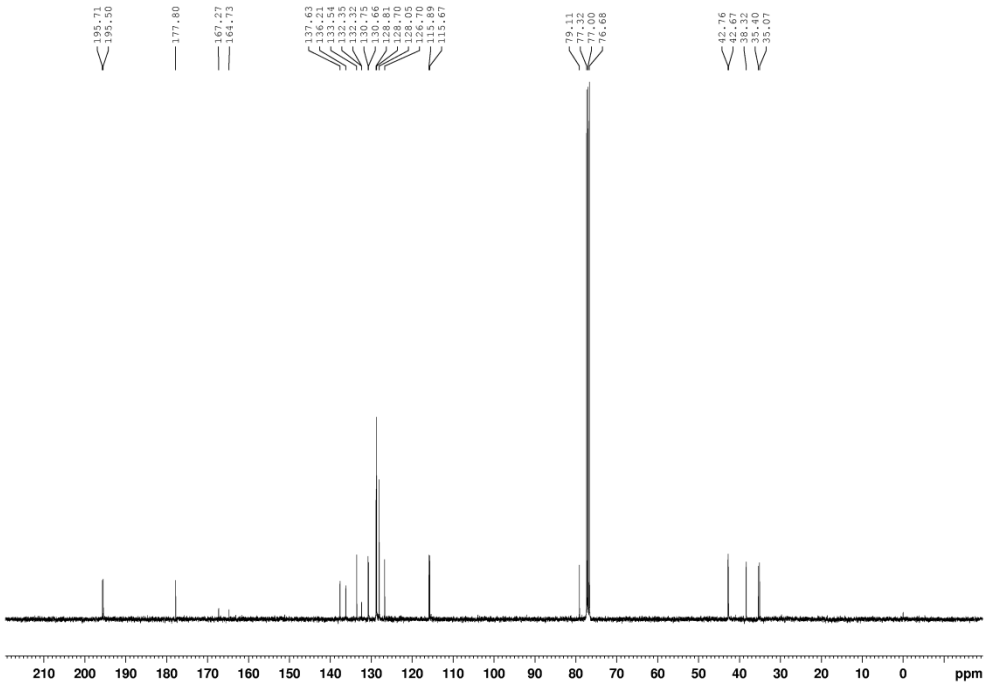

O
O
O
O
Br
Bn
Ph

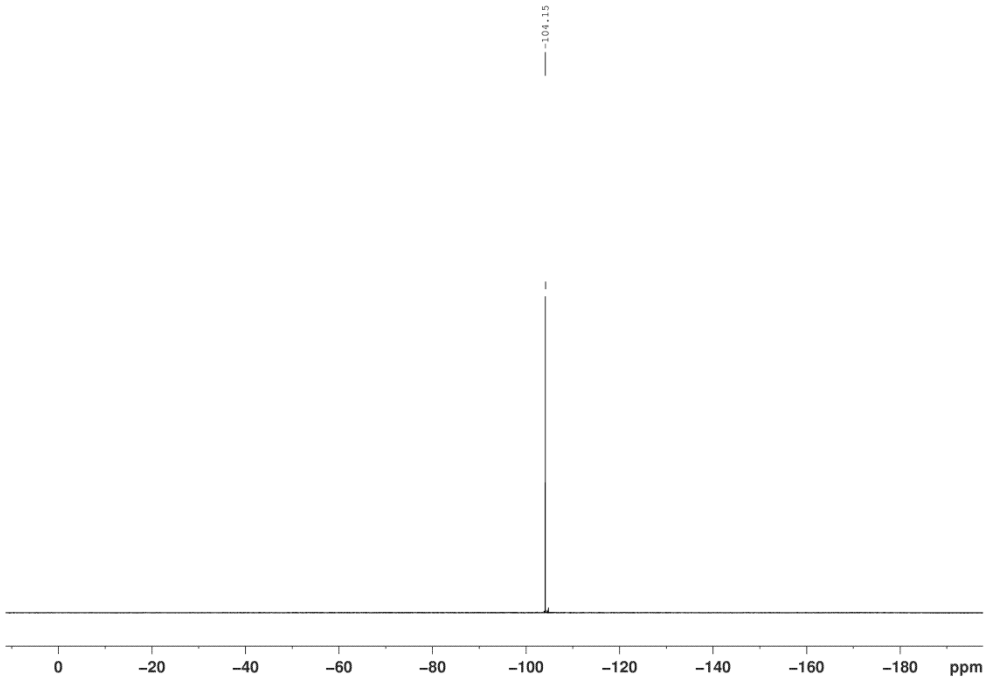

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.99
1.97
2.98
2.41
1.96
2.96
1.00
1.08
1.05
1.02
1.09
2.06
1.03
1.10


196.13
195.71
177.72
137.61
136.22
134.59
133.55
131.97
129.52
128.84
128.80
128.73
128.71
128.06
126.74
79.16
77.32
77.00
76.69
42.73
42.66
38.29
35.44
35.13
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
O
O
O
F
Bn
Ph


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.95 1.98 1.03 2.03 6.83 1.00 1.02 1.02 2.00 2.00 1.02 1.13


195.71
195.50
177.80
167.27
164.73
137.63
136.21
133.54
132.35
132.22
130.75
130.66
128.81
128.70
128.05
126.70
115.89
115.67
79.11
77.32
77.00
76.68
42.76
42.67
38.42
35.40
35.07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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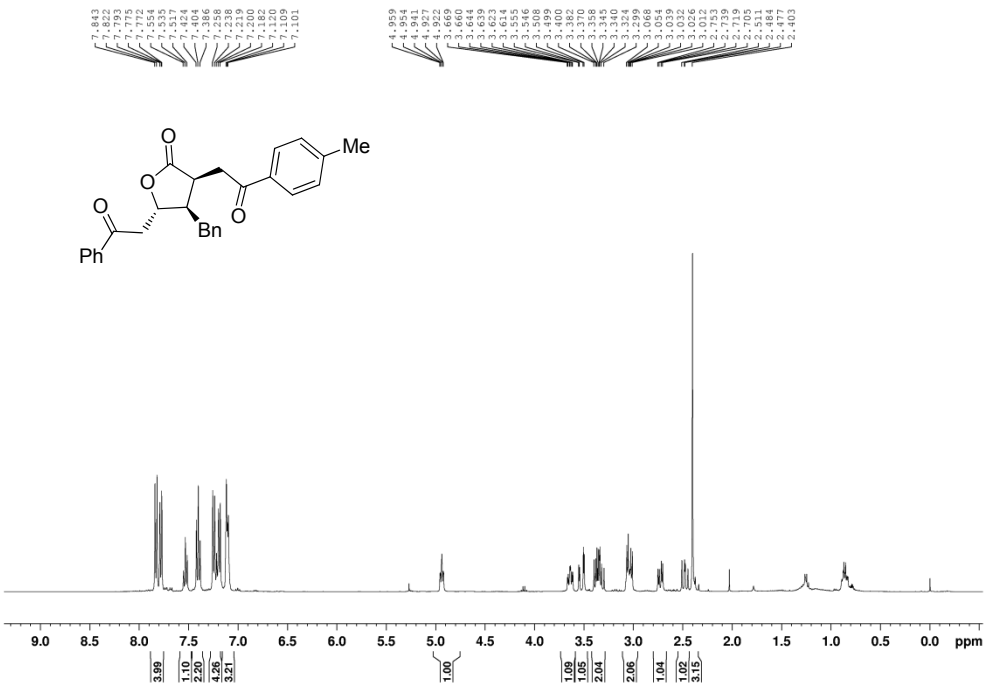
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm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7.772
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7.517
7.414
7.404
7.386
7.258
7.238
7.239
7.200
7.182
7.120
7.109
7.101
4.959
4.954
4.941
4.927
4.922
3.669
3.660
3.644
3.639
3.623
3.614
3.555
3.546
3.538
3.499
3.400
3.382
3.370
3.348
3.345
3.340
3.324
3.299
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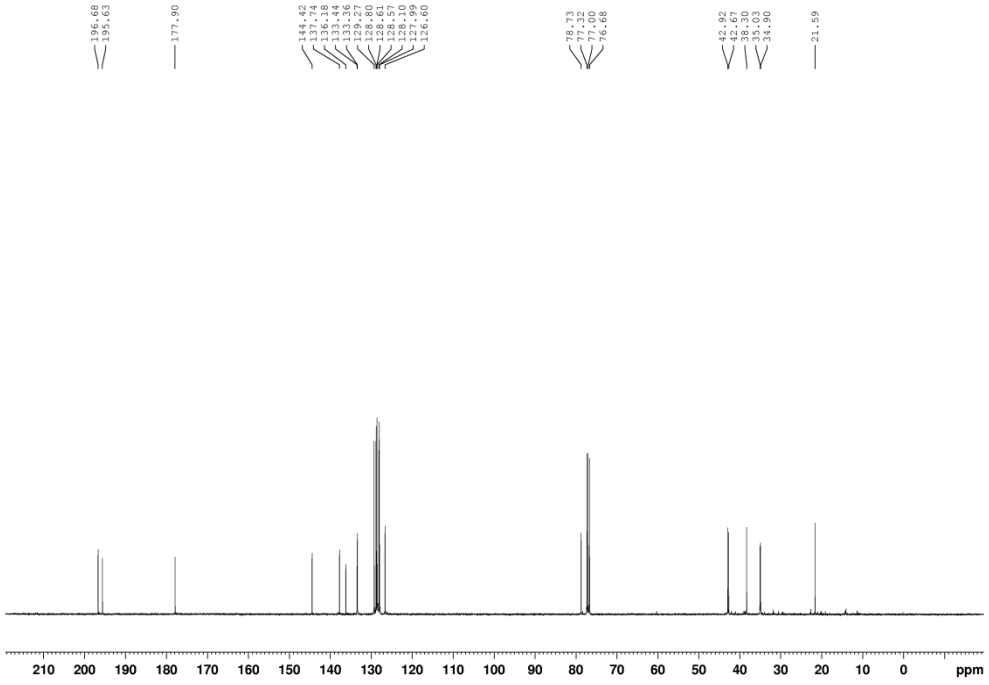
3.054
3.039
3.032
3.026
3.022
2.753
2.739
2.719
2.705
2.701
2.484
2.477
2.403
O
O
O
O
Me
Bn
Ph
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.99
1.10
2.20
4.26
3.21
1.00
1.09
1.05
2.04
2.06
1.04
1.02
3.15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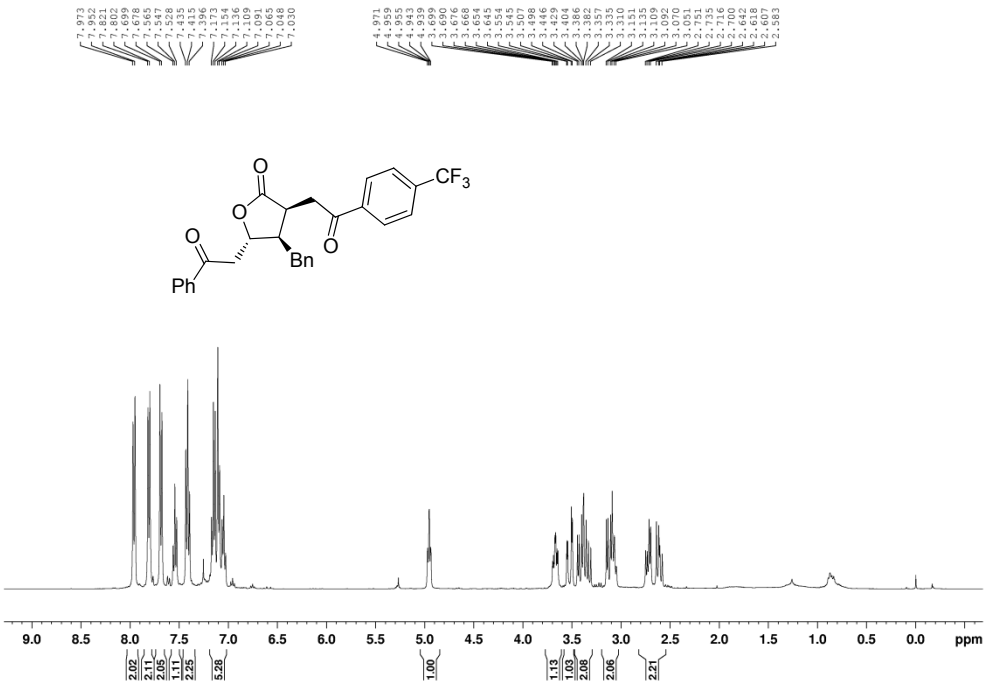
195.63
177.90
144.42
137.74
135.18
133.44
133.36
129.27
128.80
128.61
128.57
128.10
127.99
126.60
78.73
77.32
77.00
76.68
42.92
42.67
38.00
35.03
34.90
21.59
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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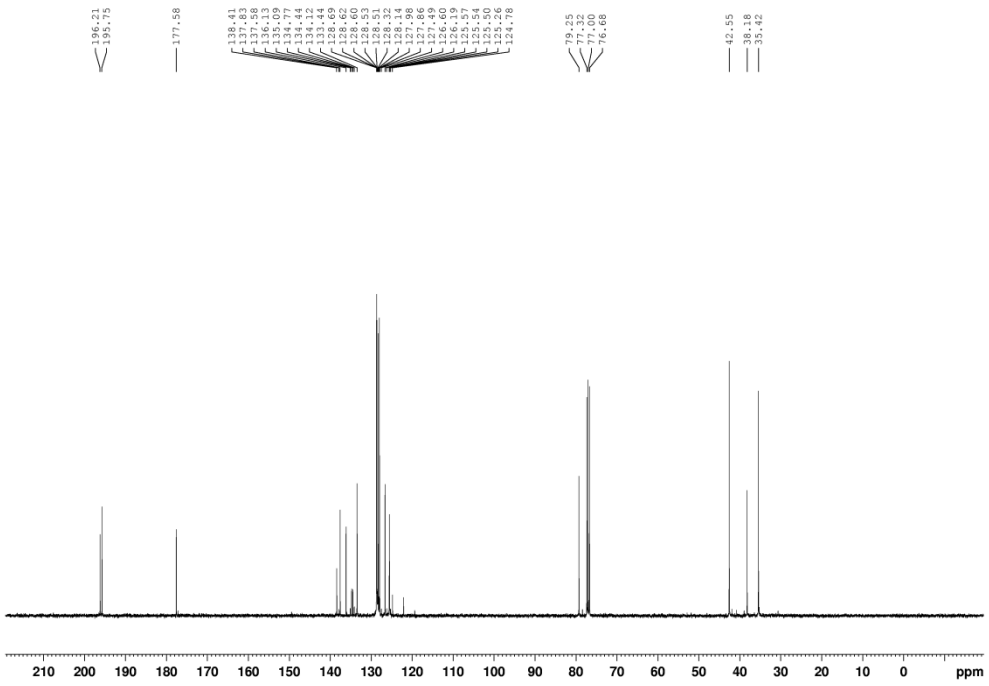
7.154
7.136
7.109
7.091
7.045
7.048
7.030
4.971
4.959
4.955
4.943
4.939
3.699
3.690
3.676
3.668
3.654
3.645
3.554
3.545
3.507
3.498
3.446
3.429
3.404
3.386
3.382
3.357
3.335
3.310
3.151
3.135
3.109
3.092
3.070
3.051
2.751
2.735
2.716
2.700
2.642
2.618
2.607
2.583
O
O
O
O
CF3
Bn
Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
2.11
2.05
1.11
2.25
5.28
1.00
1.13
1.03
2.08
2.06
2.21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195.75
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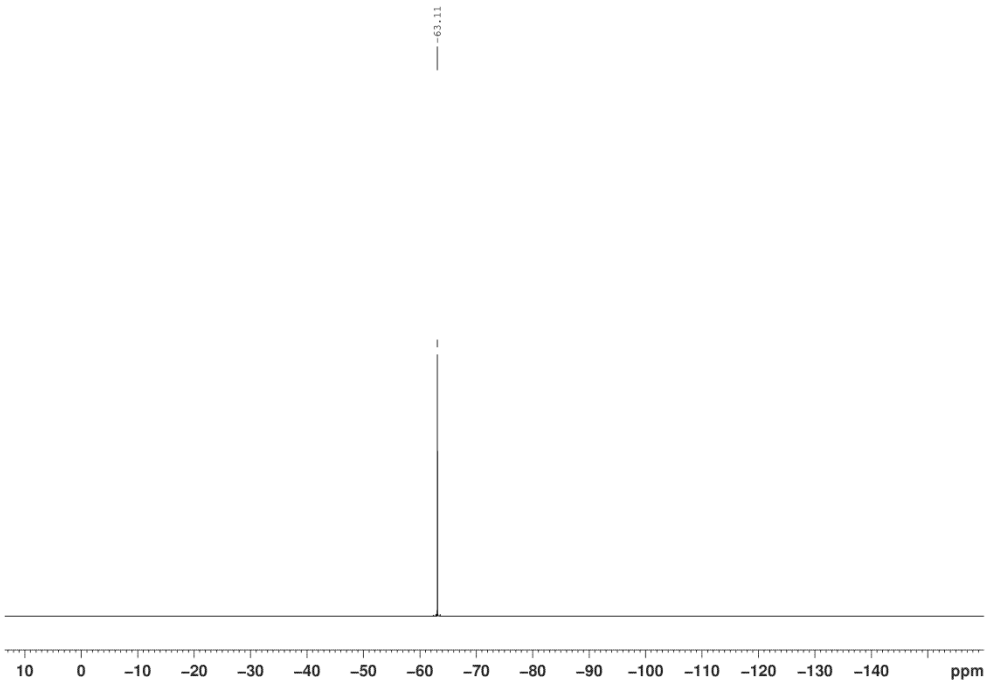
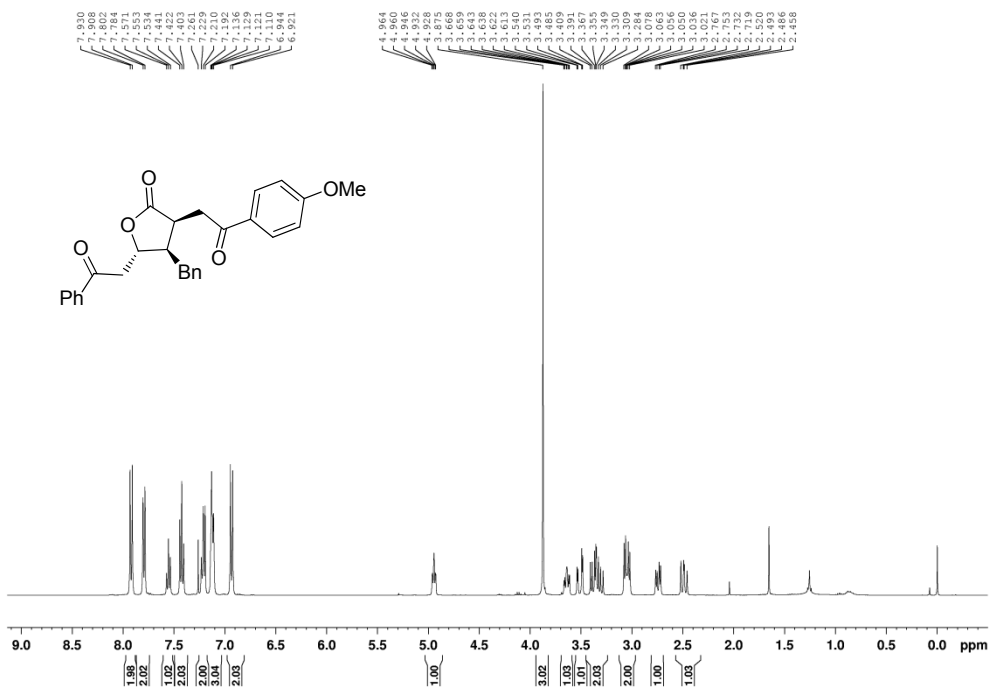
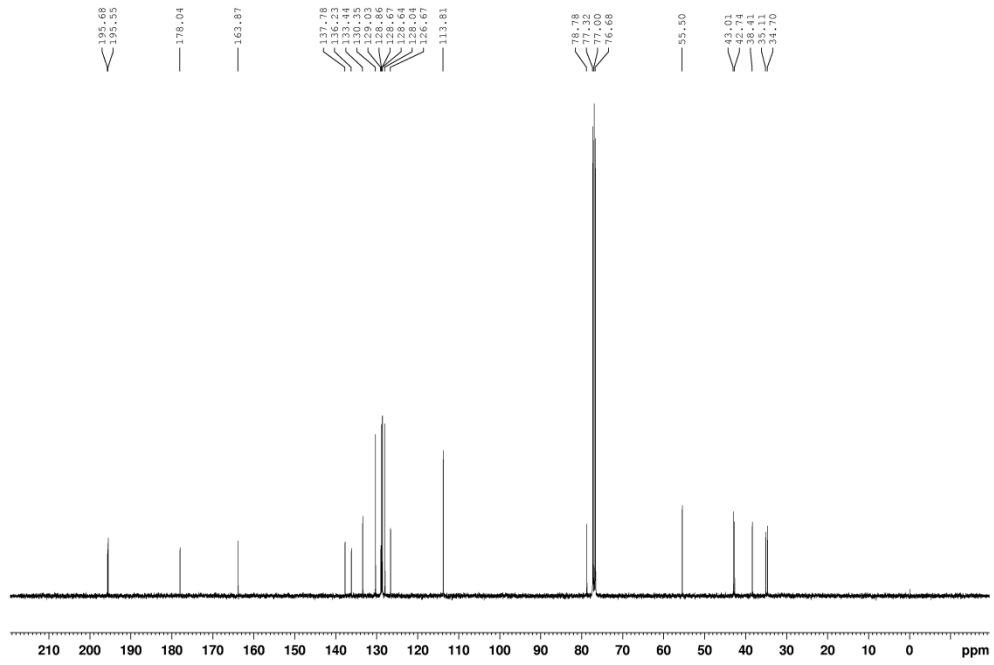
137.83
137.58
136.13
135.09
134.77
134.44
134.12
133.44
128.69
128.62
128.60
128.53
128.51
128.32
128.14
128.04
127.98
127.86
127.49
126.60
126.19
125.57
125.54
125.50
125.26
124.78
79.25
77.32
77.00
76.68
42.55
38.18
35.42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


-63.11
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 ppm


OMe
O
O
O
O
Bn
Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.441
7.996
7.993
7.974
7.954
7.905
7.893
7.884
7.873
7.827
7.809
7.641
7.624
7.606
7.604
7.591
7.586
7.572
7.567
7.548
7.457
7.438
7.419
7.358
7.202
7.183
7.165
7.121
7.120
7.081
7.063
7.045
4.993
4.978
4.964
3.734
3.726
3.715
3.695
3.687
3.679
3.646
3.520
3.498
3.472
3.466
3.426
3.404
3.387
3.130
3.115
3.102
3.097
3.088
3.073
3.014
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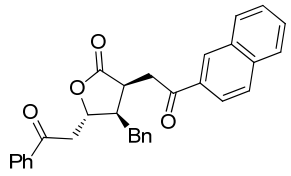
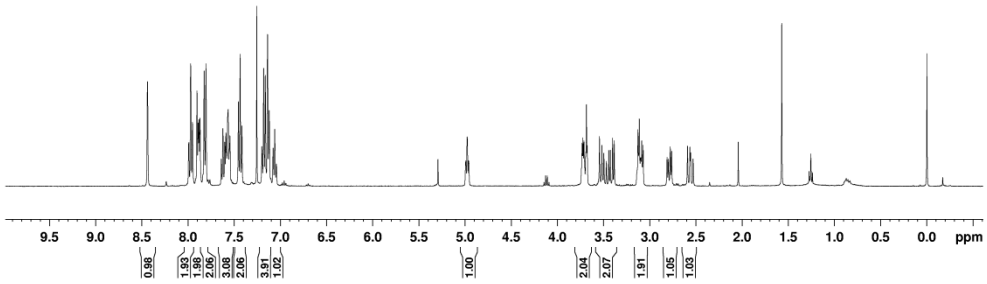
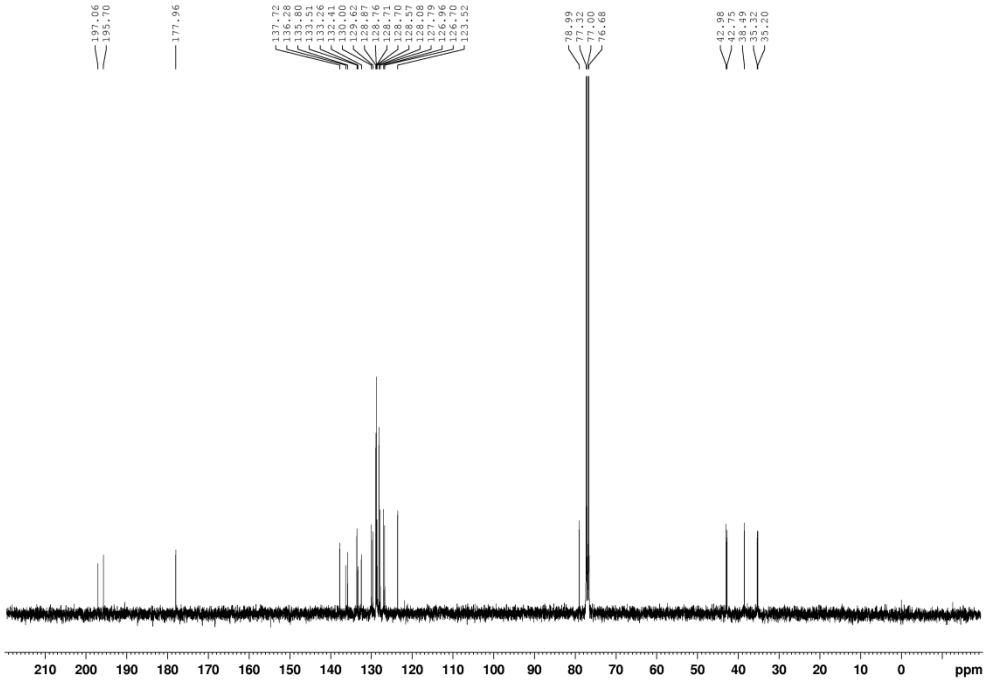
2.780
2.765
2.593
2.567
2.558
2.532


O
O
O
O
Bn
Ph


9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
1.83
1.98
2.06
3.08
2.06
3.91
1.02
1.00
2.04
2.07
1.91
1.05
1.03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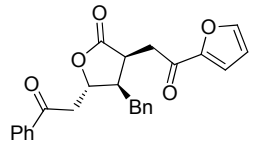
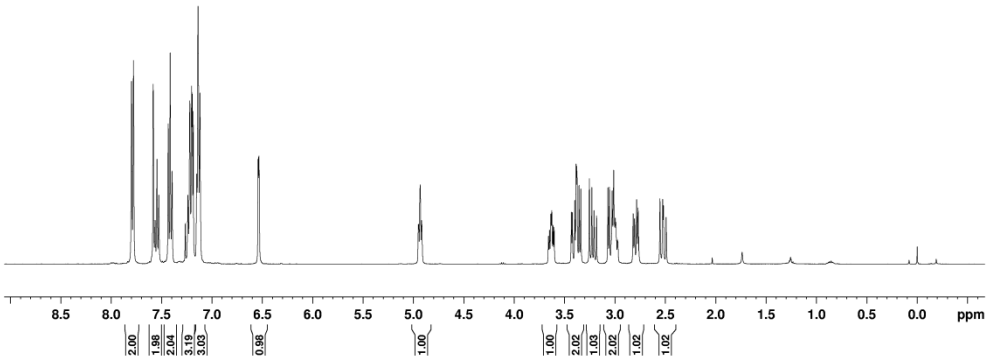
195.70
177.96
137.72
136.28
135.60
133.51
133.26
132.41
130.00
129.62
128.87
128.76
128.71
128.70
128.37
128.08
127.79
126.96
126.70
123.52
78.99
77.32
77.00
76.68
42.98
42.75
38.49
35.32
35.20
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


O
O
O
O
O
Bn
Ph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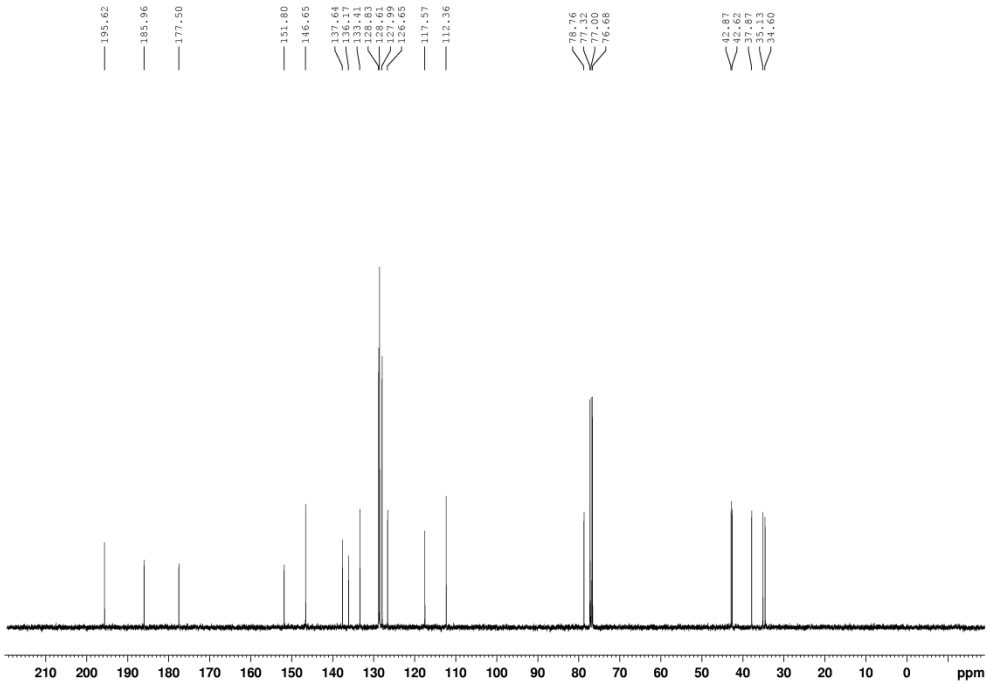
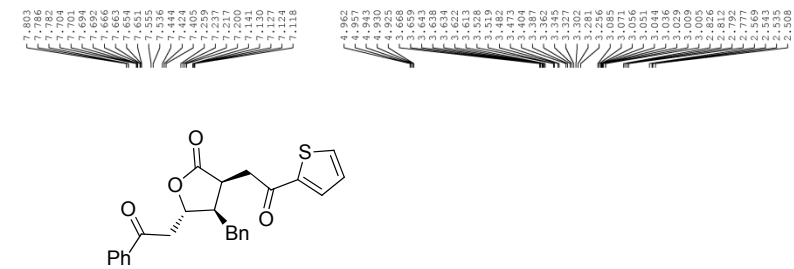
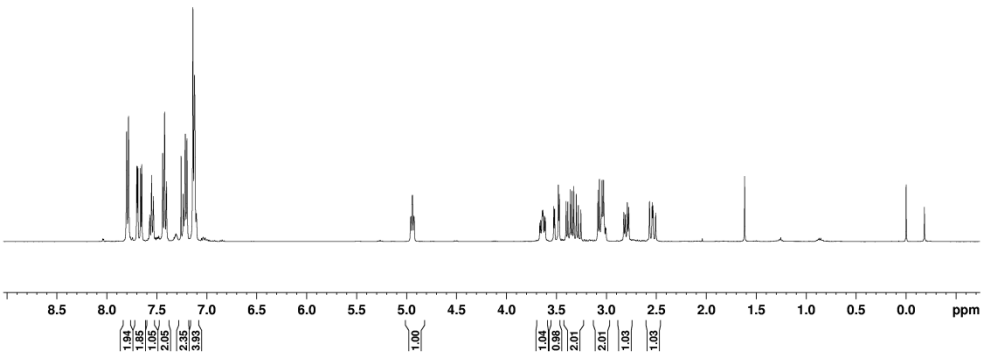
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.00
1.98
2.04
3.19
9.03
0.98
1.00
1.00
2.02
1.03
2.02
1.02
1.02


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm




O
O
S
O
O
Bn
Ph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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.94
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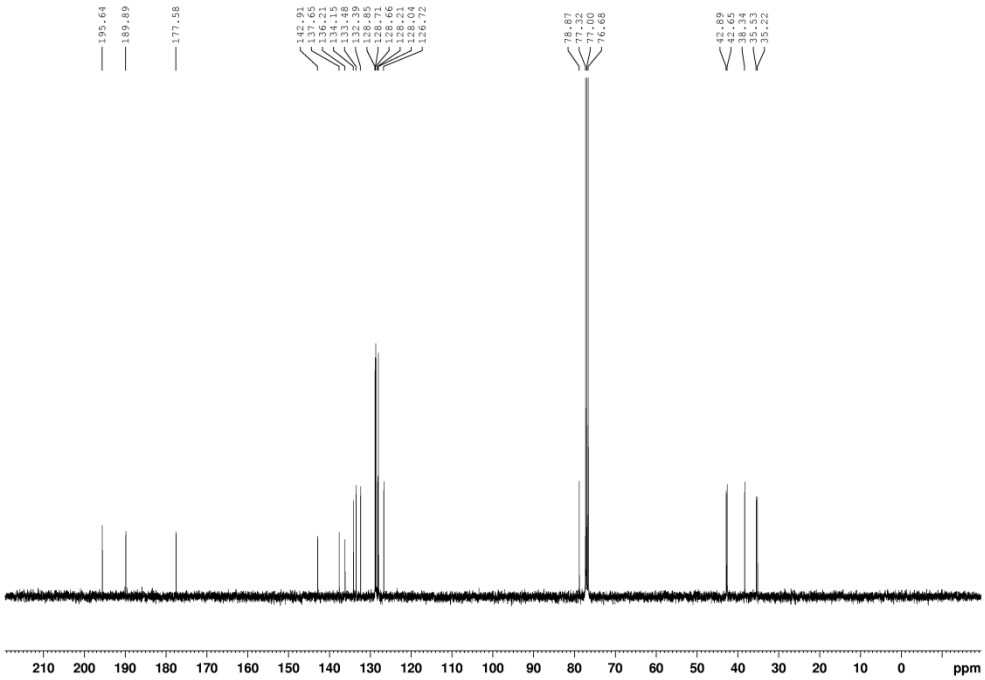
1.05
2.05
2.35
3.93
1.00
1.04
0.98
2.01
2.01
1.03
1.03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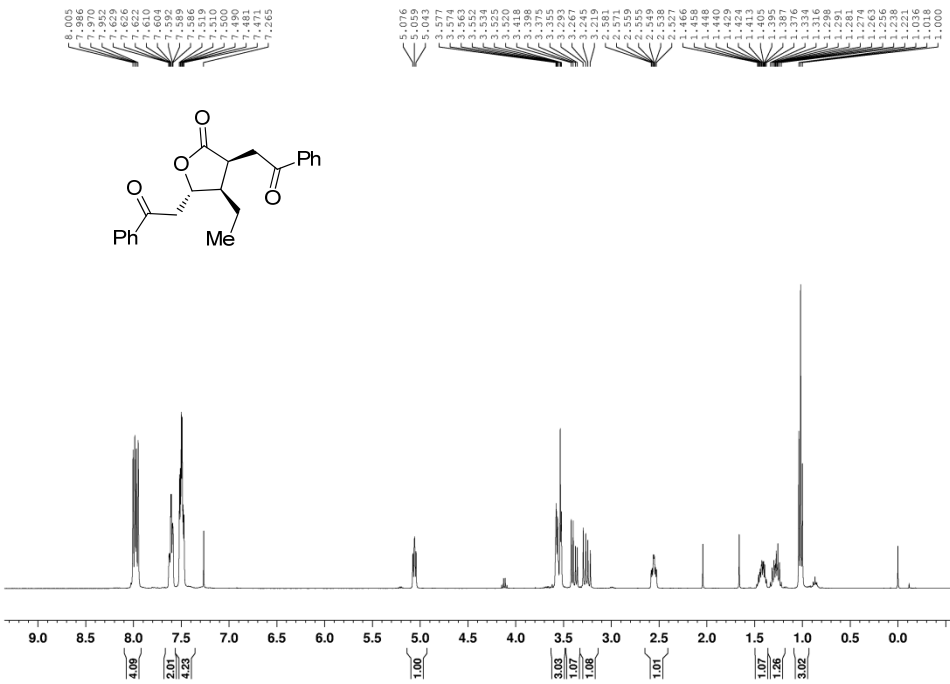
189.89
177.58
142.91
137.65
136.21
134.15
133.48
132.39
129.45
128.71
128.66
128.21
128.04
126.72
78.87
77.32
77.00
76.68
42.89
42.65
38.34
35.53
35.22
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.622
7.610
7.604
7.592
7.589
7.586
7.519
7.510
7.500
7.490
7.481
7.471
7.265
5.076
5.059
5.043
3.577
3.574
3.560
3.552
3.534
3.525
3.520
3.418
3.398
3.375
3.355
3.293
3.267
3.245
3.219
2.581
2.571
2.559
2.555
2.549
2.538
2.527
1.466
1.458
1.448
1.440
1.429
1.424
1.413
1.405
1.395
1.387
1.376
1.334
1.316
1.298
1.291
1.281
1.274
1.263
1.256
1.238
1.221
1.036
1.018
1.000
O
O
O
O
Ph
Ph
Me
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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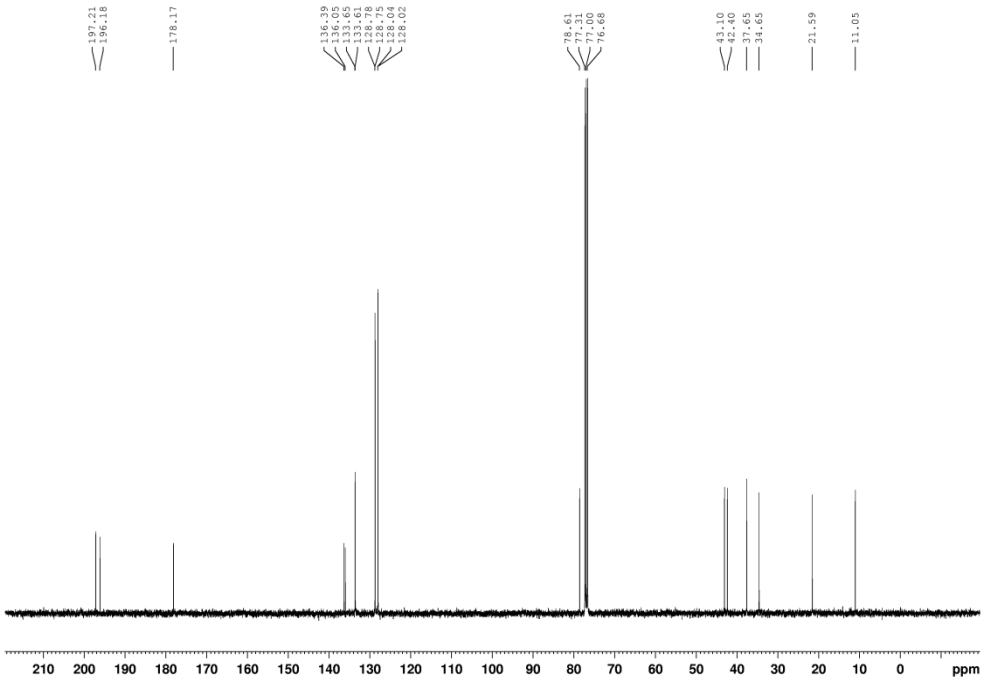
2.01
4.23
1.00
3.03
1.07
1.08
1.01
1.07
1.26
3.02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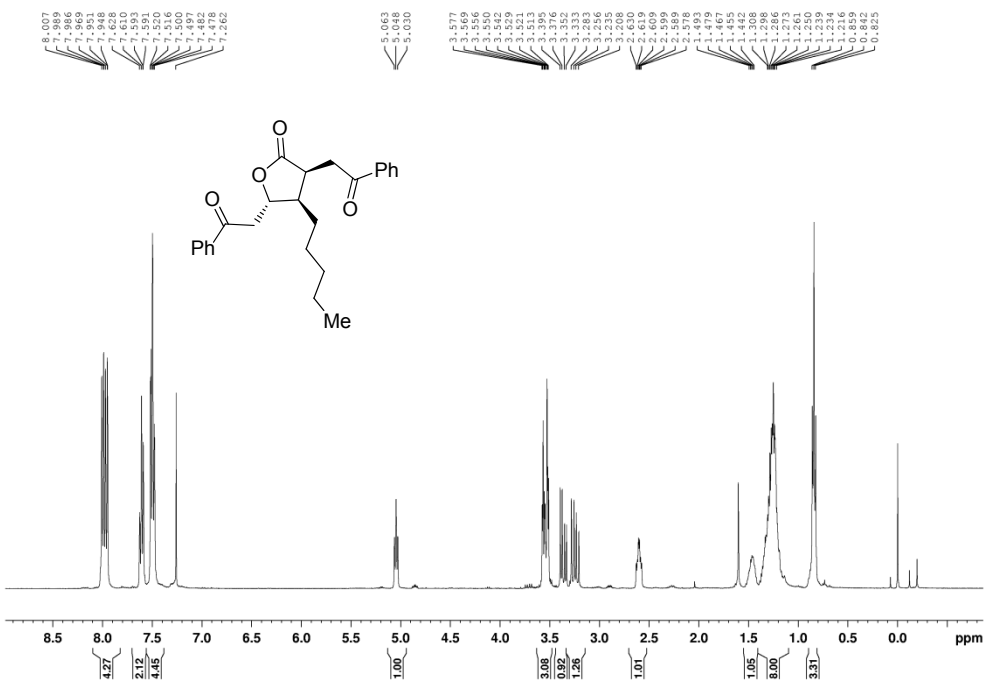
196.18
178.17
136.39
136.05
133.65
133.61
128.78
128.75
128.04
128.02
78.61
77.31
77.00
76.68
43.10
42.40
37.65
34.65
21.59
11.05
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.610
7.593
7.591
7.520
7.516
7.500
7.497
7.492
7.478
7.262
5.063
5.048
5.030
3.577
3.569
3.556
3.550
3.542
3.529
3.521
3.513
3.395
3.376
3.352
3.333
3.283
3.256
3.235
3.008
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2.619
2.609
2.599
2.589
2.578
1.493
1.479
1.467
1.465
1.442
1.308
1.298
1.286
1.273
1.261
1.250
1.239
1.234
1.214
0.859
0.842
0.825
O
O
Ph
O
O
Ph
Me
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
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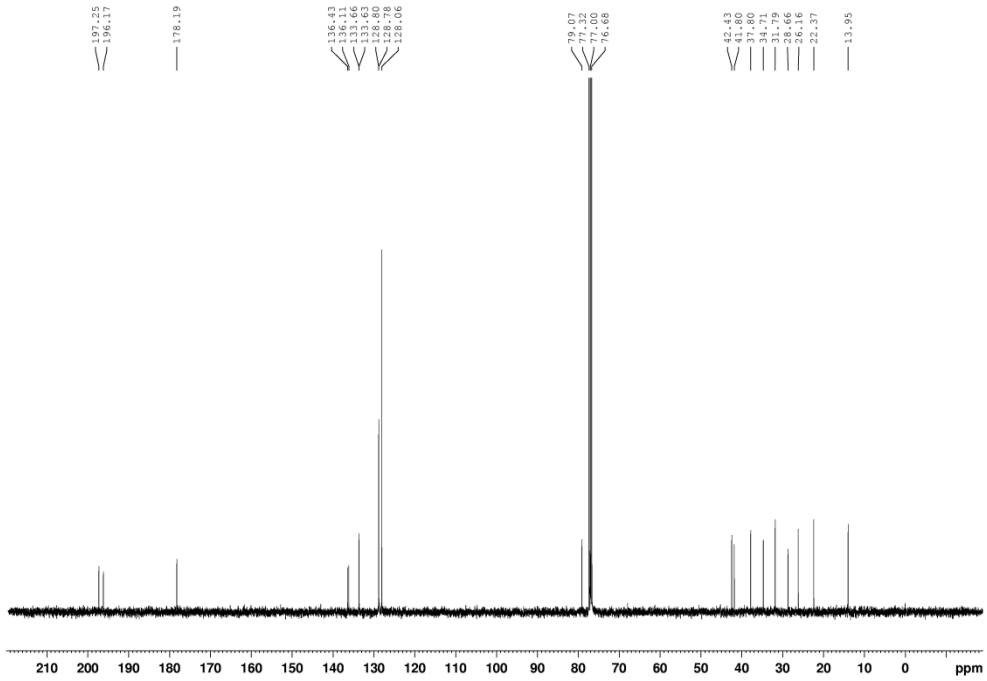
4.45
1.00
3.08
0.92
1.26
1.01
1.05
8.00
3.31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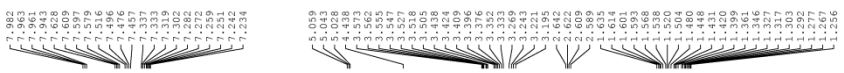
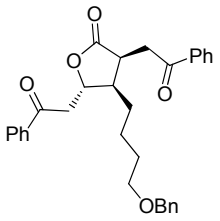
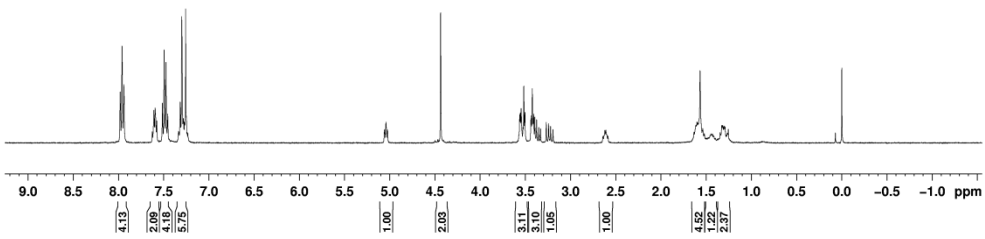
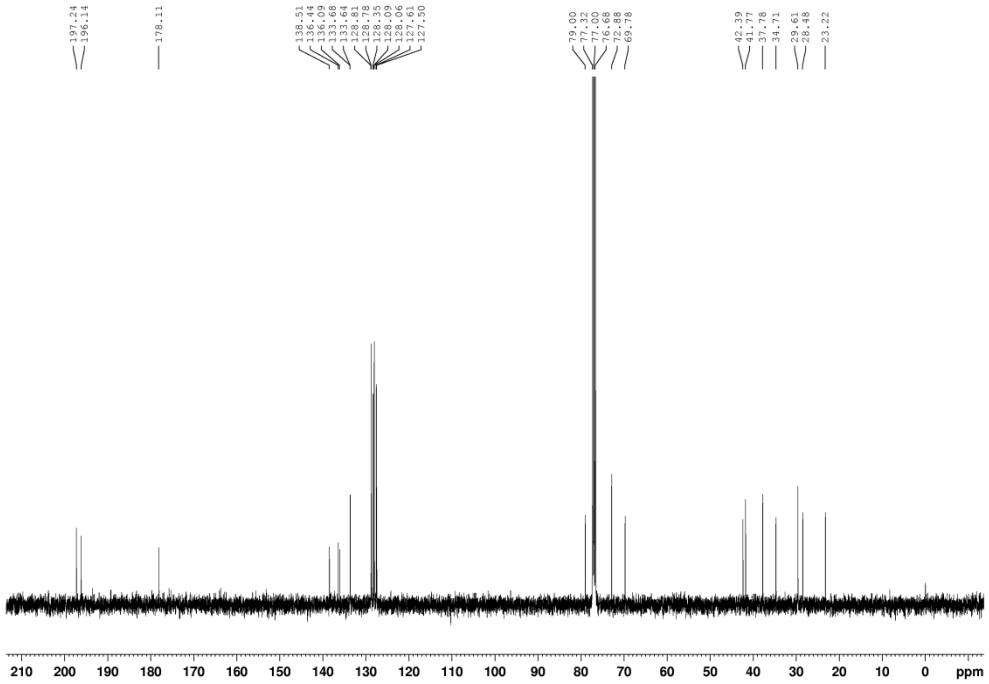
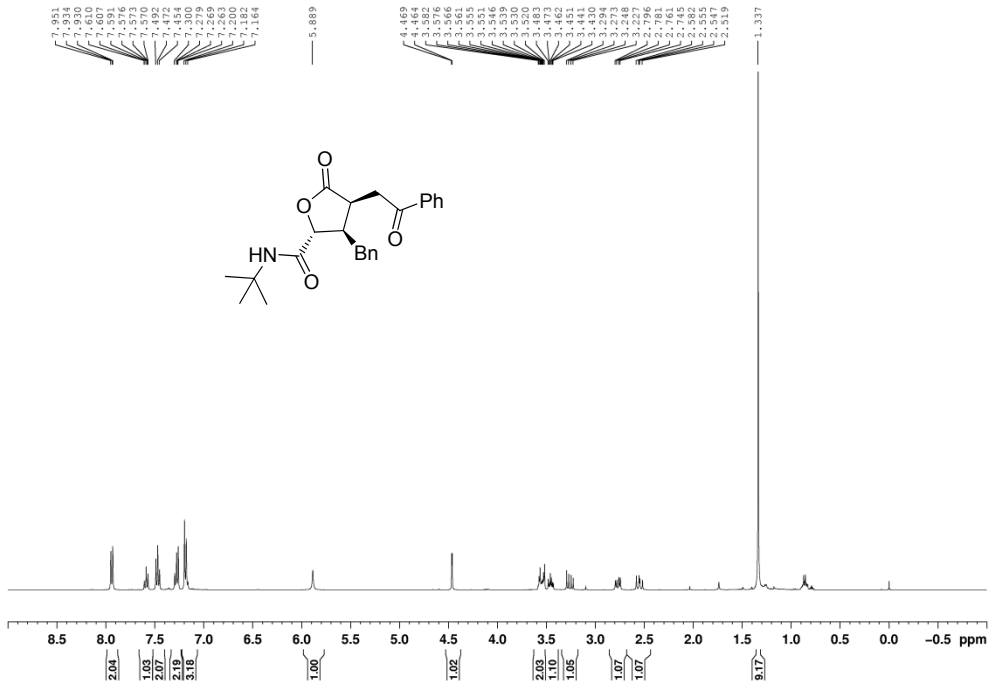
196.17
178.19
136.43
136.11
133.66
133.63
128.80
128.78
128.06
79.07
77.32
77.00
76.68
42.43
41.80
37.80
34.71
31.79
28.66
26.16
22.37
13.95
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm




O
O
Ph
O
O
Ph
OBn


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0 ppm
4.13
2.09
4.16
5.75
1.00
2.03
3.11
3.10
1.05
1.00
4.52
1.22
2.37


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.573
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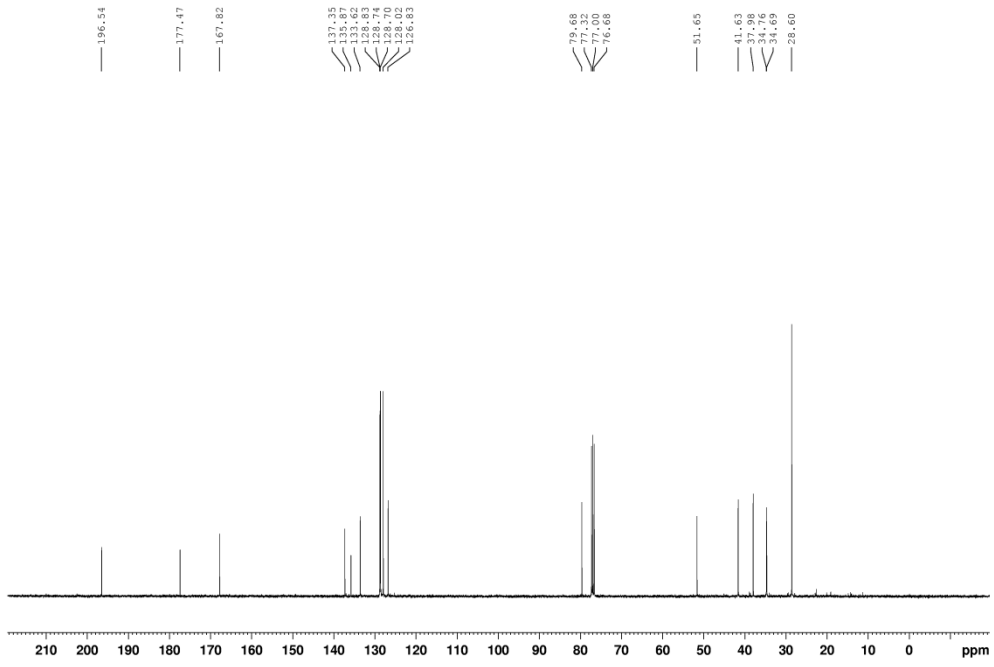
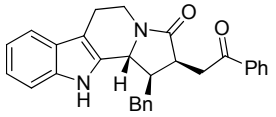
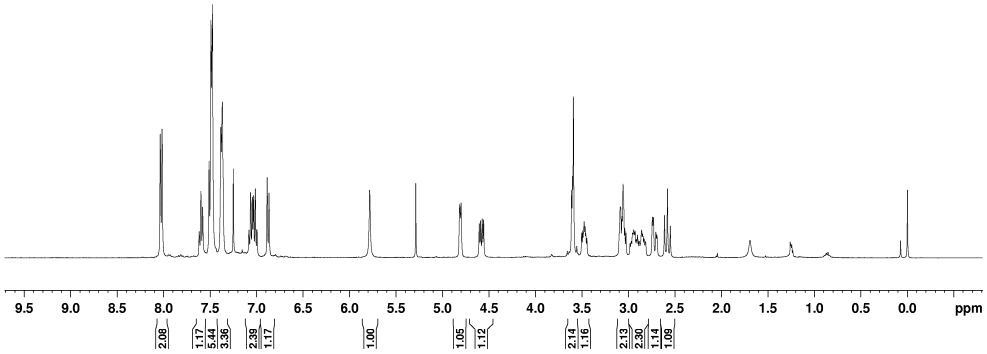
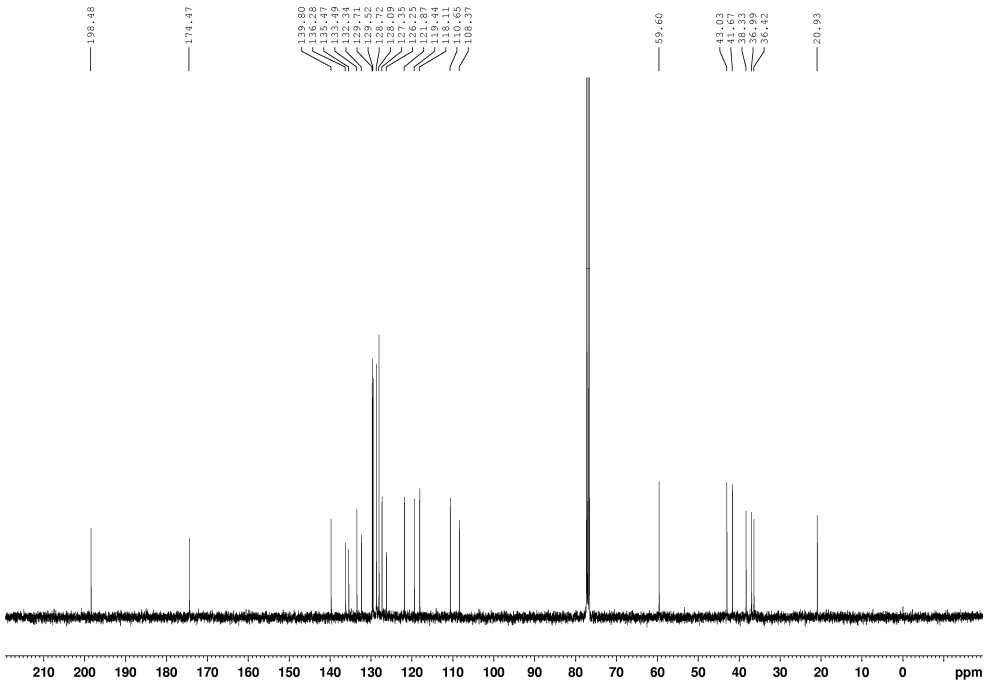
7.492
7.472
7.454
7.300
7.279
7.269
7.263
7.180
7.164
5.889
4.469
4.464
4.462
3.576
3.566
3.561
3.555
3.551
3.546
3.539
3.530
3.520
3.483
3.473
3.462
3.451
3.441
3.430
3.294
3.273
3.248
3.227
2.796
2.781
2.761
2.745
2.582
2.555
2.547
2.519
1.337
O
O
Ph
O
HN
Bn
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
2.04
1.03
2.07
2.19
3.18
1.00
1.02
2.03
1.10
1.05
1.07
1.07
9.17


196.54
177.47
167.82
137.35
135.87
133.62
128.83
128.74
128.70
128.02
126.83
79.68
77.32
77.00
76.68
51.65
41.63
37.98
34.76
34.69
28.60
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.036
8.017
7.598
7.580
7.511
7.490
7.473
7.384
7.368
7.253
7.067
7.048
7.034
7.015
6.868
6.868
5.785
4.817
4.803
4.607
4.592
4.575
4.566
4.543
3.604
3.594
3.505
3.494
3.479
3.468
3.460
3.449
3.090
3.085
3.071
3.060
3.041
3.028
2.970
2.960
2.955
2.945
2.931
2.920
2.914
2.904
2.887
2.865
2.850
2.844
2.836
2.830
2.820
2.744
2.733
2.706
2.695
2.614
2.582
2.551

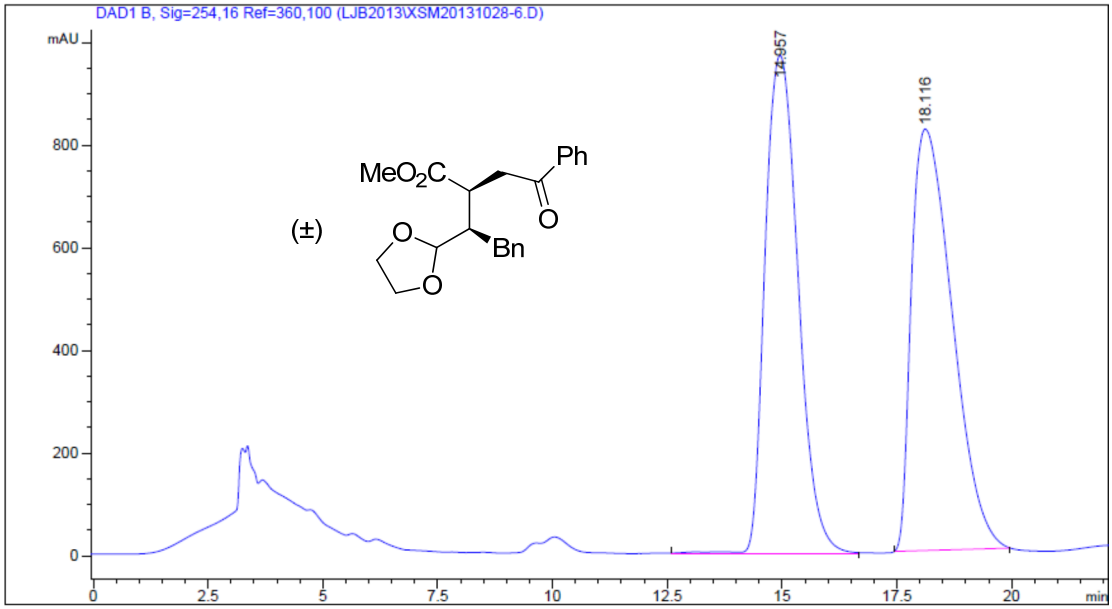

O
N
O
Ph
N
H
H
Bn


9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.08
1.17
5.44
3.36
2.39
1.17
1.00
1.05
1.12
2.14
1.16
2.13
2.30
1.14
1.08

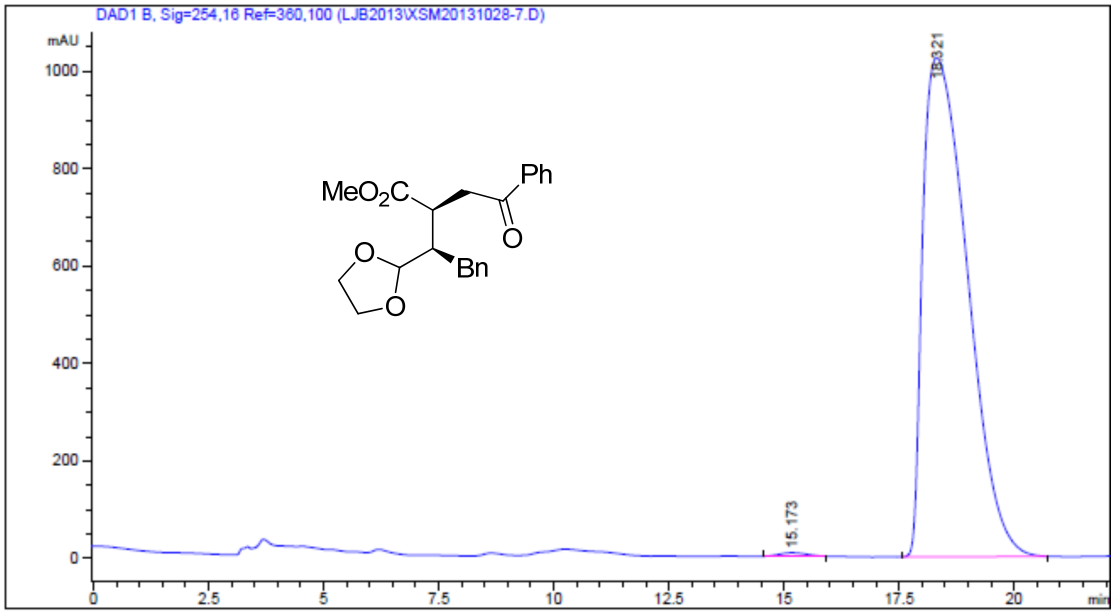

198.48
174.47
139.80
136.28
135.47
133.49
132.34
132.31
129.52
128.72
128.09
127.35
126.75
121.87
119.44
118.11
110.65
108.37
59.60
43.03
41.67
38.33
36.99
36.42
20.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

# 7. HPLC Traces

**4a**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

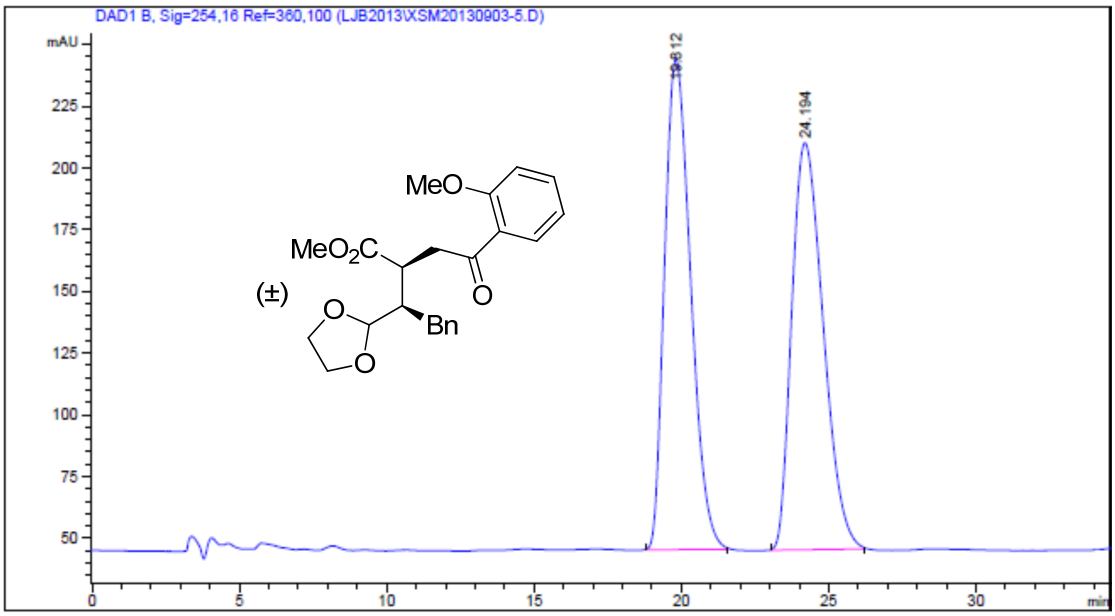



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 14.957 | 4.88738e4 | 971.79749 | 49.04 |
| 2 | DAD 254.16 nm | 18.116 | 5.07881e4 | 821.73883 | 50.96 |

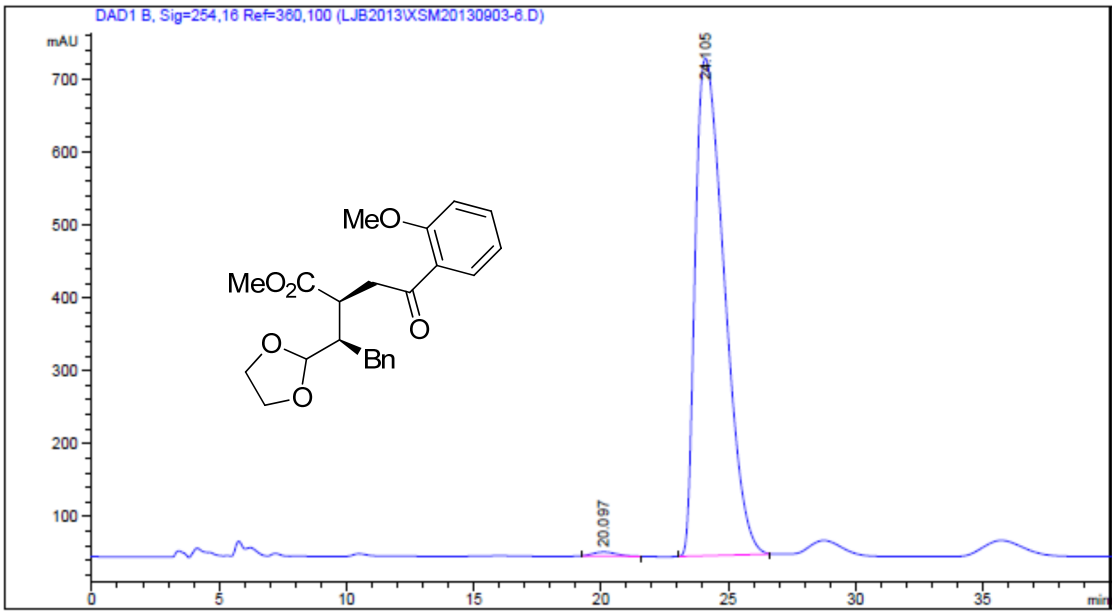



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 15.173 | 328.97687 | 7.63727 | 0.47 |
| 2 | DAD 254.16 nm | 18.321 | 6.89407e4 | 1025.60596 | 99.53 |

**4b**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

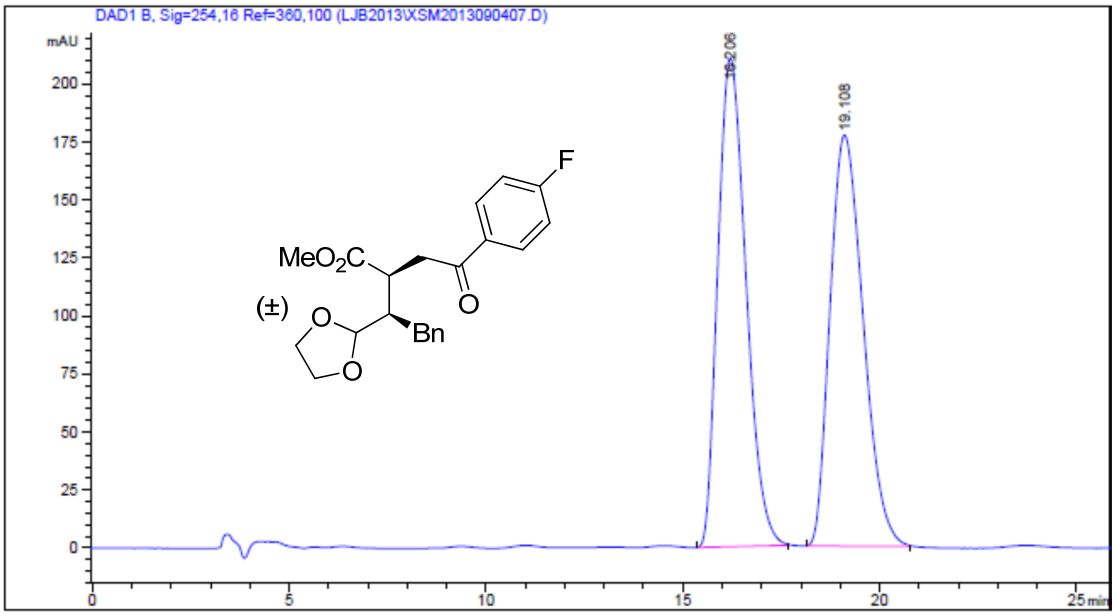



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 19.812 | 1.22204e4 | 198.87541 | 50.04 |
| 2 | DAD 254.16 nm | 24.194 | 1.22028e4 | 164.81760 | 49.96 |

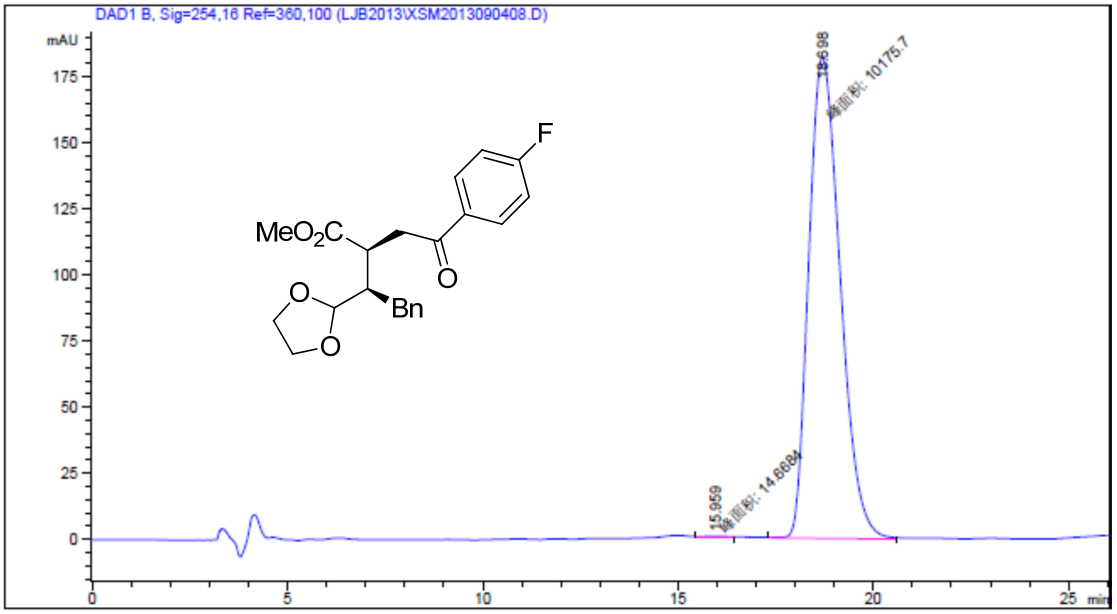



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 20.097 | 362.96405 | 5.74699 | 0.67 |
| 2 | DAD 254.16 nm | 24.105 | 5.41029e4 | 682.76758 | 99.33 |

**4c**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

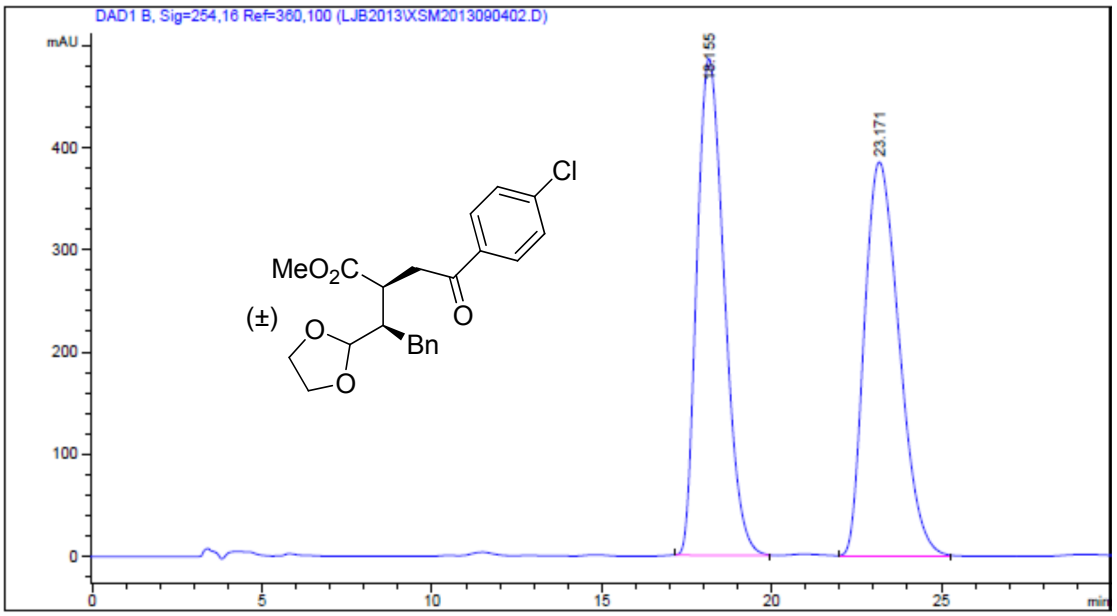



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 16.206 | 1.04704e4 | 210.78694 | 50.23 |
| 2 | DAD 254.16 nm | 19.108 | 1.03760e4 | 177.32230 | 49.77 |

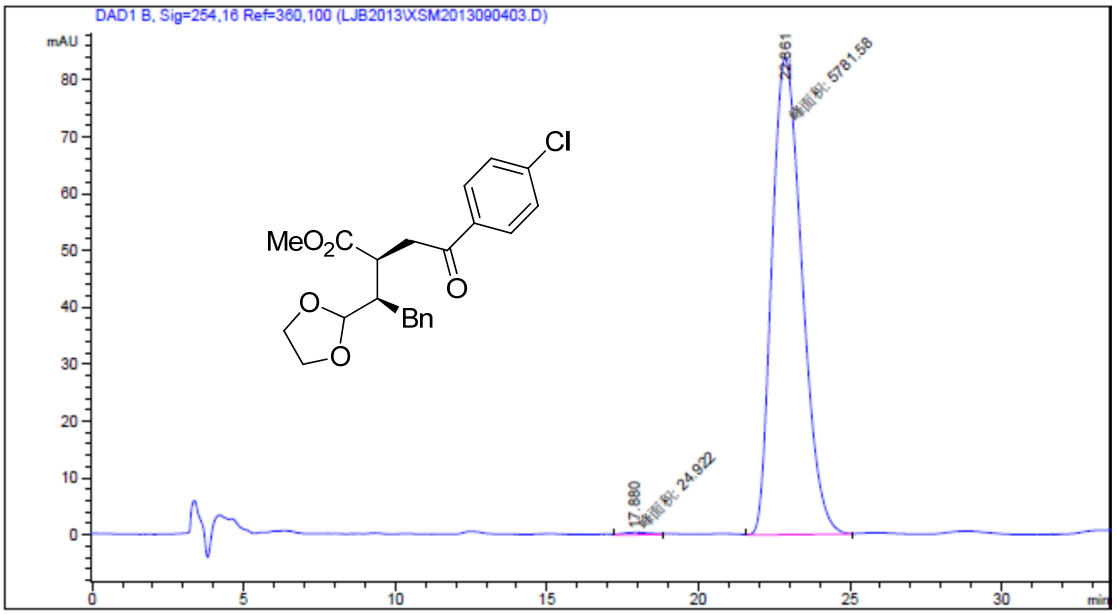



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 15.959 | 14.66843 | 3.10536e-1 | 0.14 |
| 2 | DAD 254.16 nm | 18.698 | 1.01757e4 | 181.98547 | 99.86 |

**4d**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

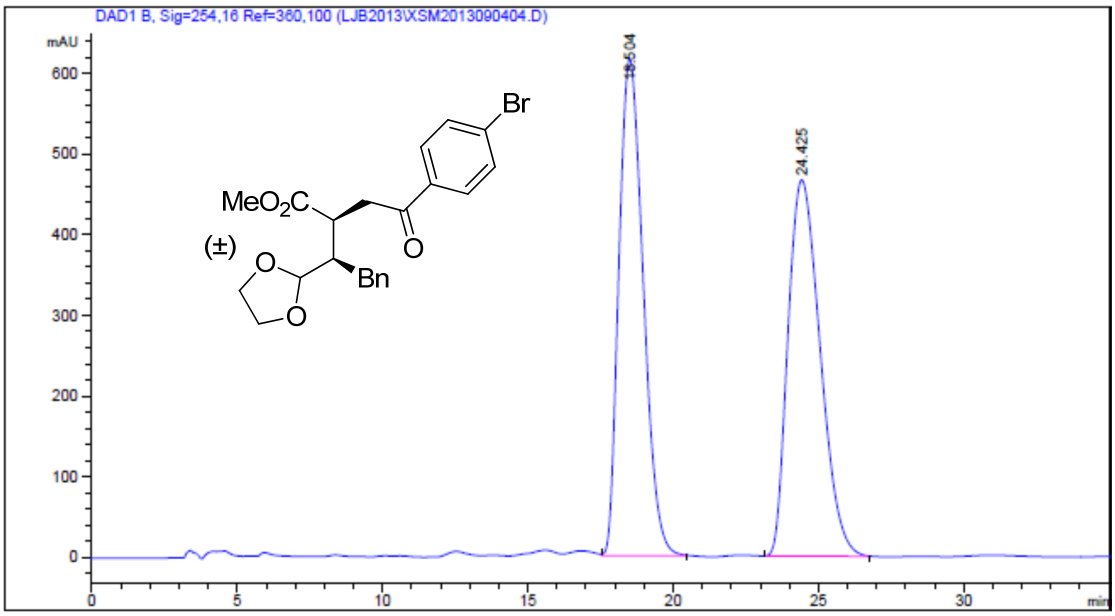



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 18.155 | 2.71440e4 | 485.63226 | 49.92 |
| 2 | DAD 254.16 nm | 23.171 | 2.72329e4 | 384.90384 | 50.08 |

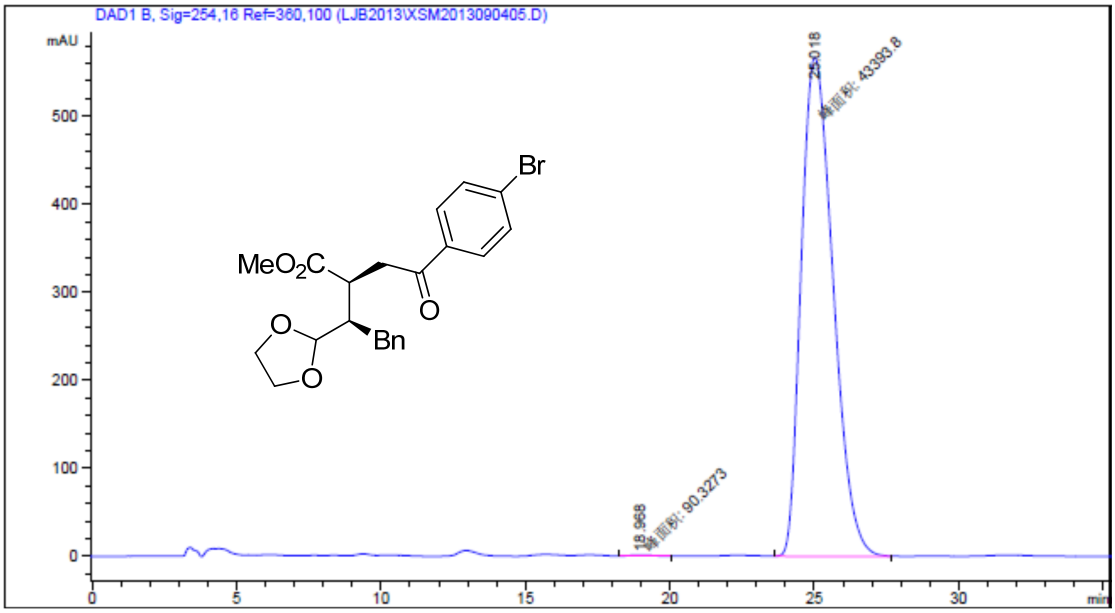



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 17.880 | 24.92195 | 3.88826e-1 | 0.43 |
| 2 | DAD 254.16 nm | 22.861 | 5781.58252 | 83.81474 | 99.57 |

**4e**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

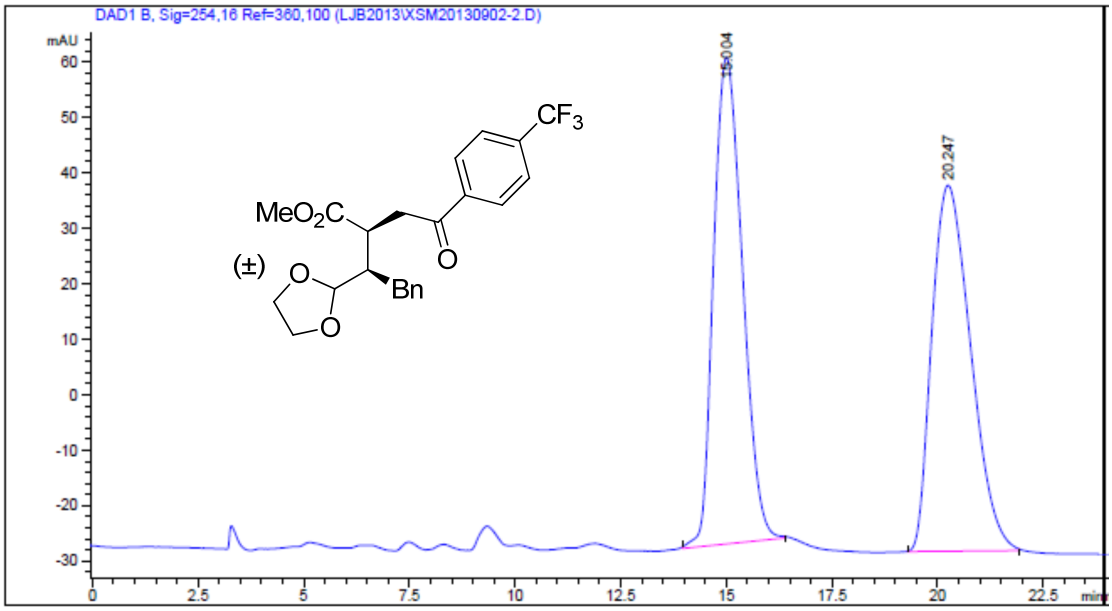



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 18.504 | 3.57017e4 | 615.98083 | 49.91 |
| 2 | DAD 254.16 nm | 24.425 | 3.58330e4 | 466.42020 | 50.09 |

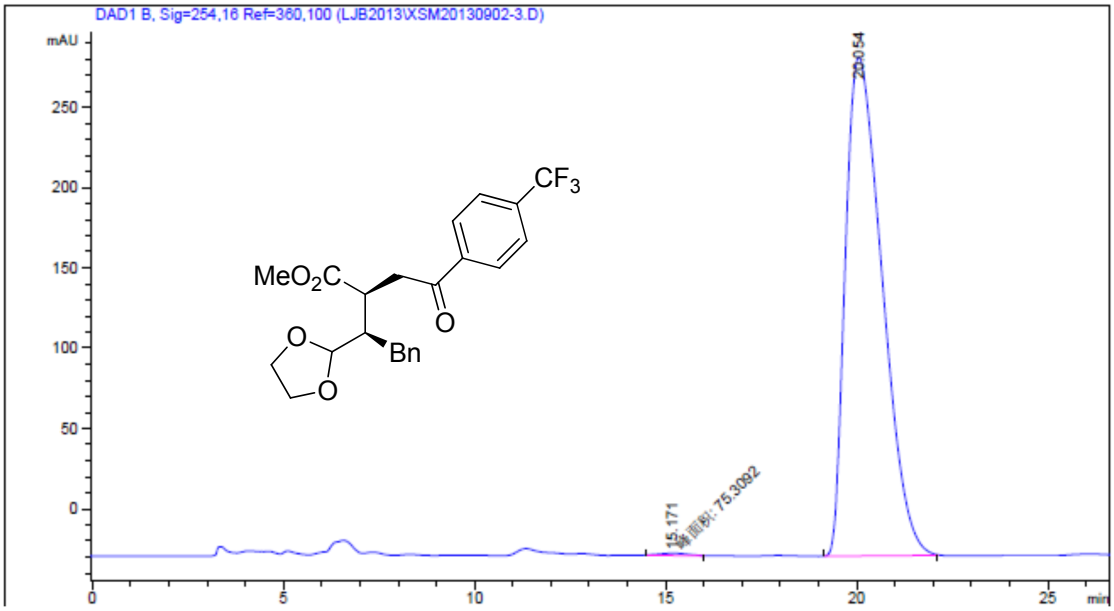



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 18.968 | 90.32731 | 1.43779 | 0.21 |
| 2 | DAD 254.16 nm | 25.018 | 4.33938e4 | 566.70764 | 99.79 |

**4f**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

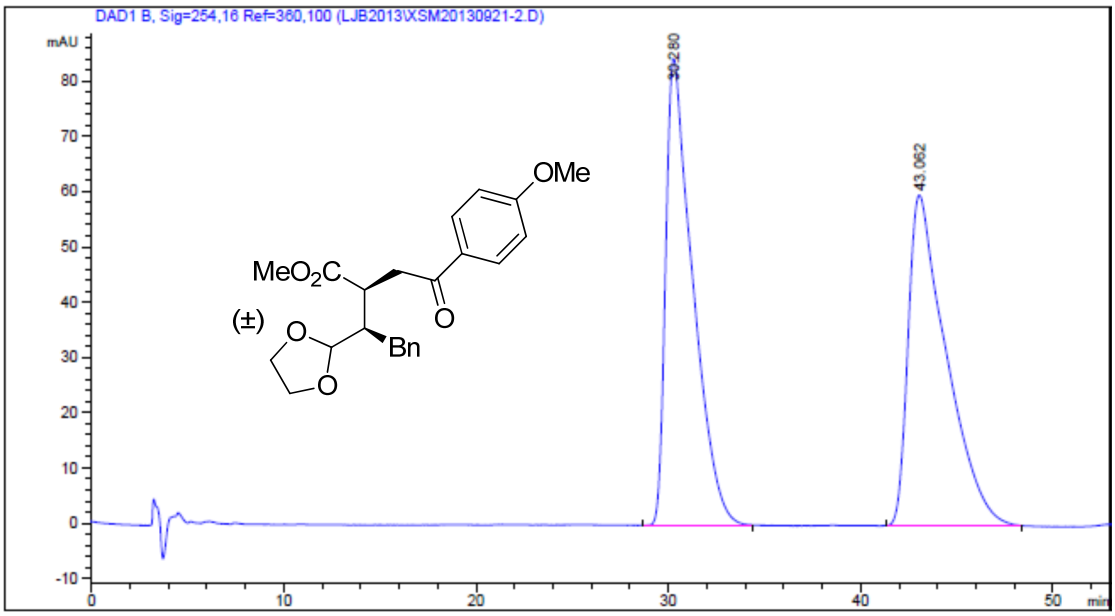



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 15.004 | 4165.52588 | 87.71129 | 49.75 |
| 2 | DAD 254.16 nm | 20.247 | 4206.86621 | 65.96428 | 50.25 |

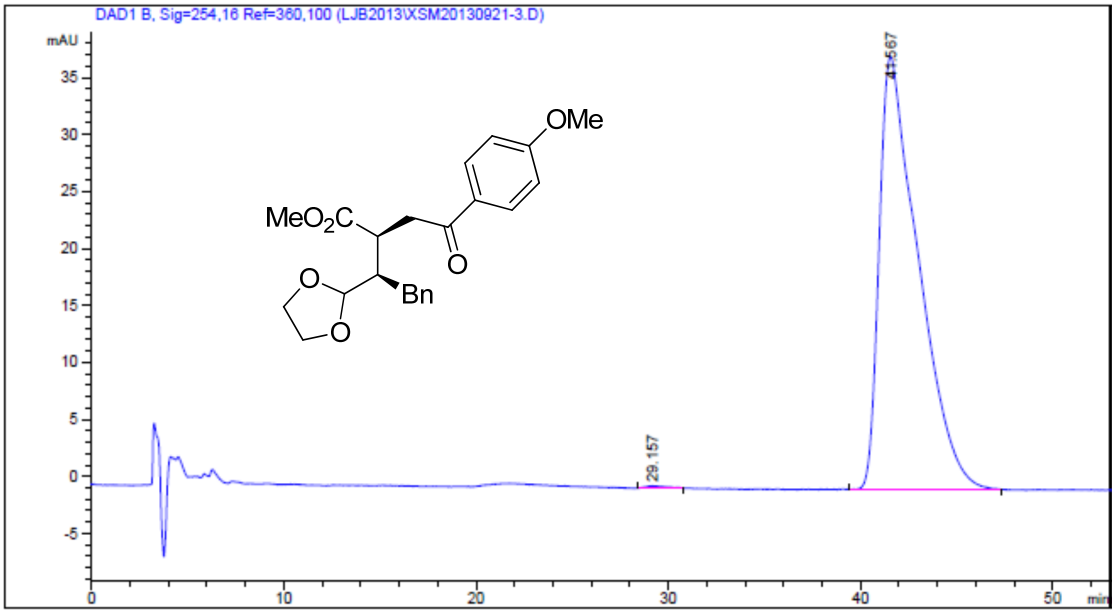



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 15.171 | 75.30917 | 1.41818 | 0.36 |
| 2 | DAD 254.16 nm | 20.054 | 2.07027e4 | 310.47659 | 99.64 |

**4g**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

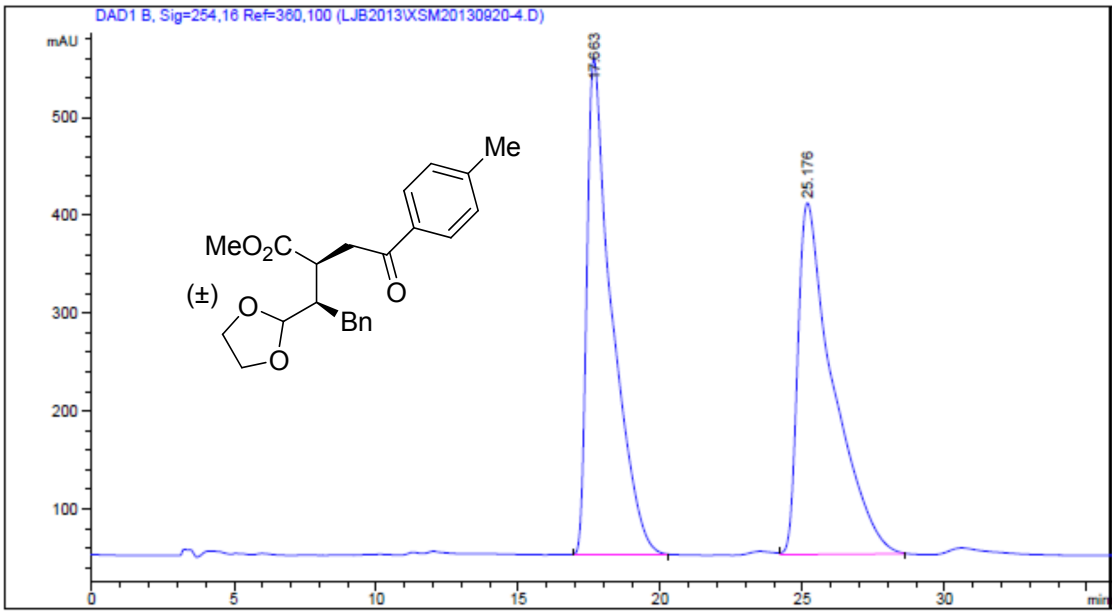



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 30.280 | 8260.16797 | 84.41898 | 50.04 |
| 2 | DAD 254.16 nm | 43.062 | 8247.27051 | 59.85088 | 49.96 |

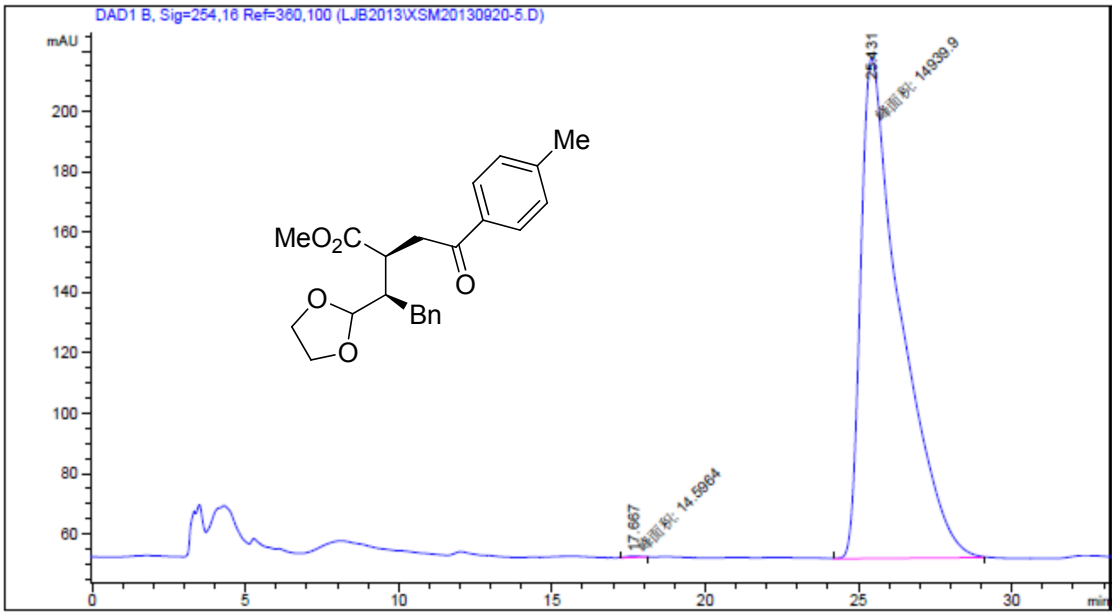



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 29.157 | 20.26181 | 2.53525e-1 | 0.38 |
| 2 | DAD 254.16 nm | 41.567 | 5350.66895 | 37.92124 | 99.62 |

**4h**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

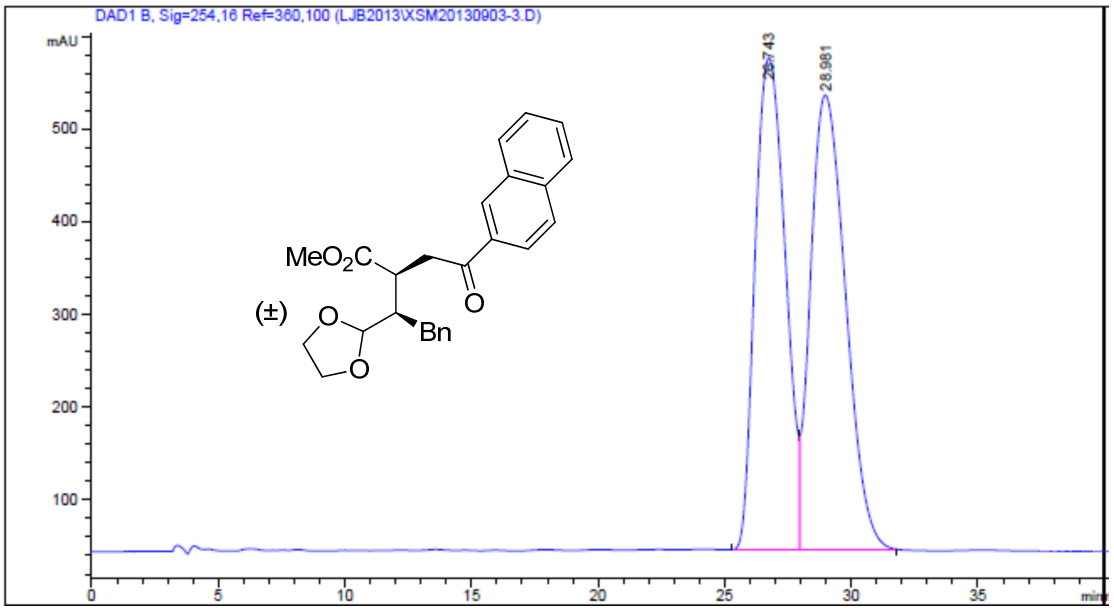



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 17.663 | 3.08912e4 | 507.39337 | 49.90 |
| 2 | DAD 254.16 nm | 25.176 | 3.10116e4 | 359.10858 | 50.10 |

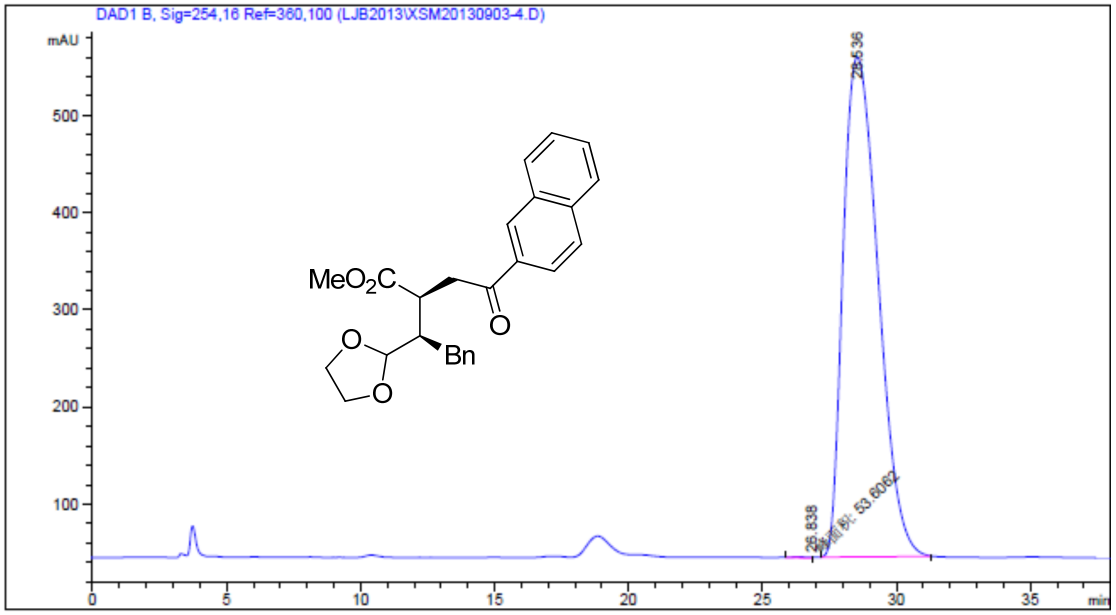



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 17.667 | 14.59635 | 4.37099e-1 | 0.09 |
| 2 | DAD 254.16 nm | 25.431 | 1.49399e4 | 165.92812 | 99.91 |

**4i**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

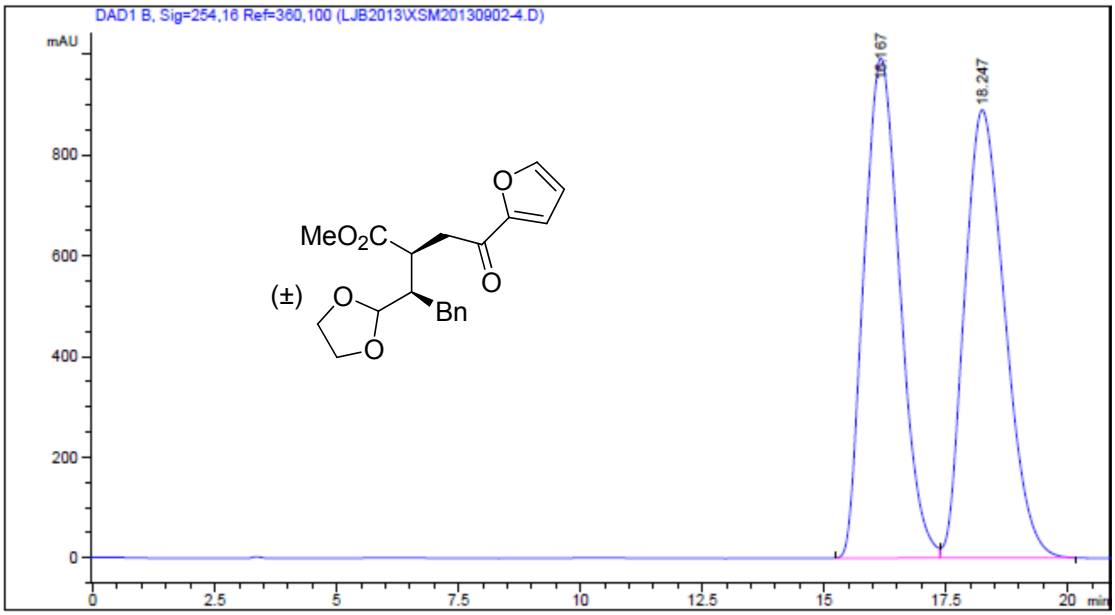



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 26.743 | 4.39159e4 | 530.34711 | 48.09 |
| 2 | DAD 254.16 nm | 28.981 | 4.73991e4 | 490.30969 | 51.91 |

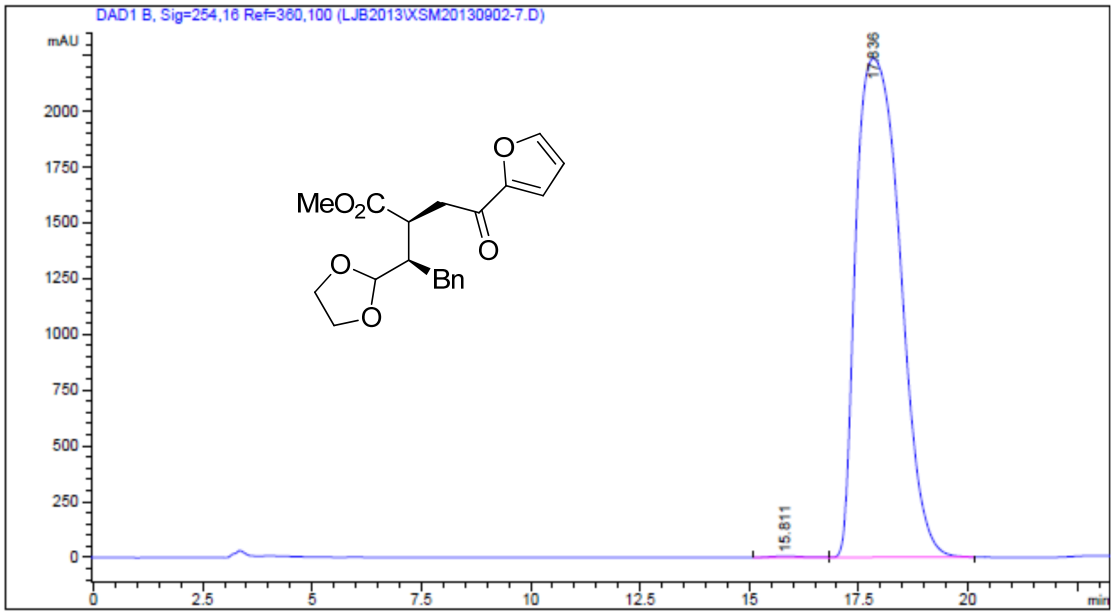



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 26.838 | 53.60617 | 1.08124 | 0.11 |
| 2 | DAD 254.16 nm | 28.536 | 4.66353e4 | 514.46033 | 99.89 |

**4j**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

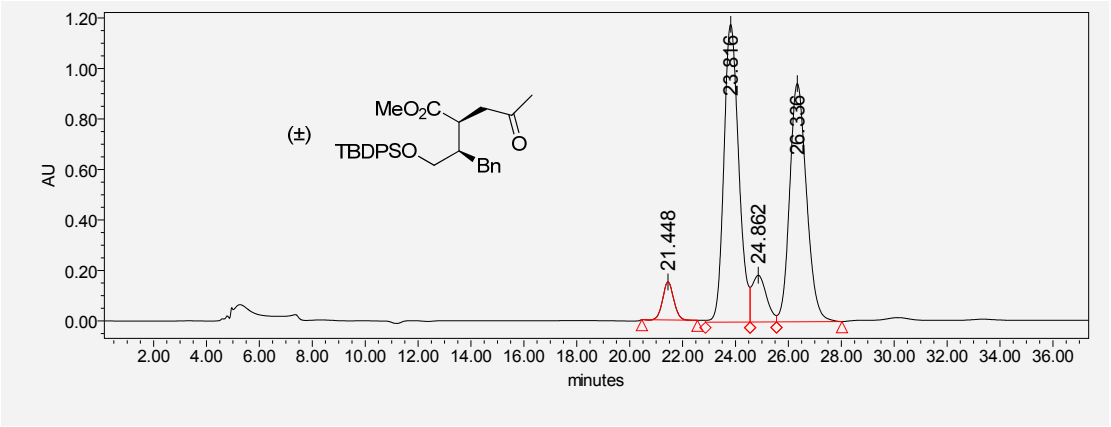



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 16.167 | 5.06221e4 | 993.38910 | 49.72 |
| 2 | DAD 254.16 nm | 18.247 | 5.11846e4 | 890.21362 | 50.28 |

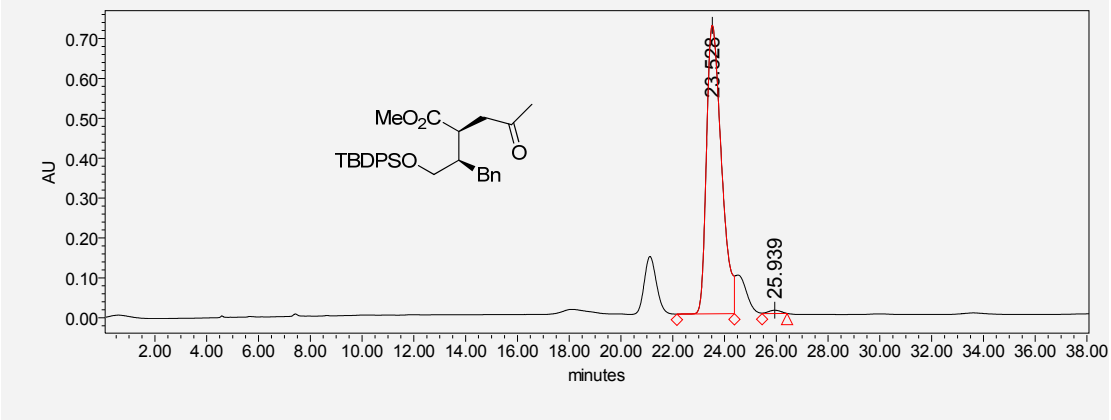



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 15.811 | 293.77023 | 5.99011 | 0.19 |
| 2 | DAD 254.16 nm | 17.836 | 1.56882e5 | 2238.78564 | 99.81 |

**4k**: Chiralpak IC Column (*n*-hexane/2-propanol = 99:1, 0.7 mL/min)

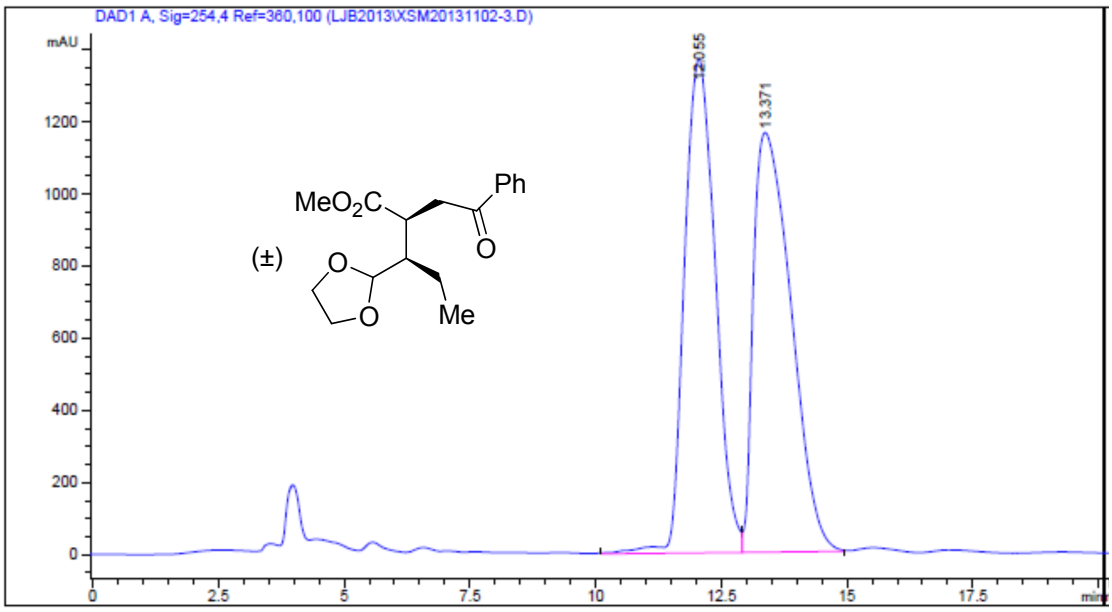



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 229.7 nm | 21.448 | 4392061 | 150188 | 4.43 |
| 2 | DAD 229.7 nm | 23.816 | 45944566 | 1178957 | 46.30 |
| 3 | DAD 229.7 nm | 24.862 | 7061156 | 184918 | 7.12 |
| 4 | DAD 229.7 nm | 26.336 | 41825848 | 943744 | 42.15 |

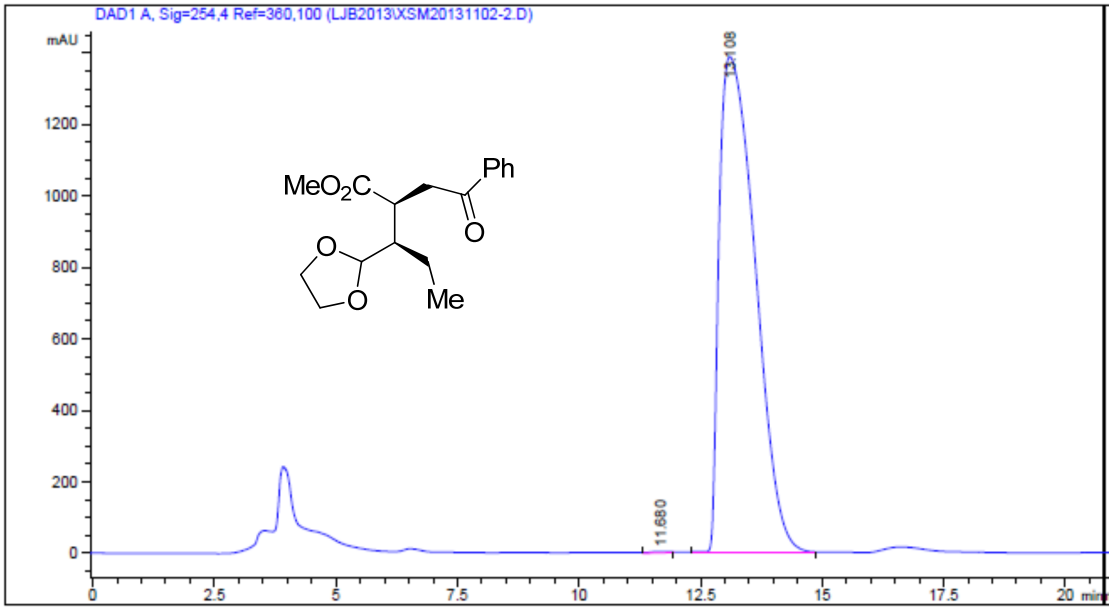



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 229.7 nm | 23.528 | 28992885 | 723119 | 99.09 |
| 2 | DAD 229.7 nm | 25.939 | 266476 | 8116 | 0.91 |

**4l**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

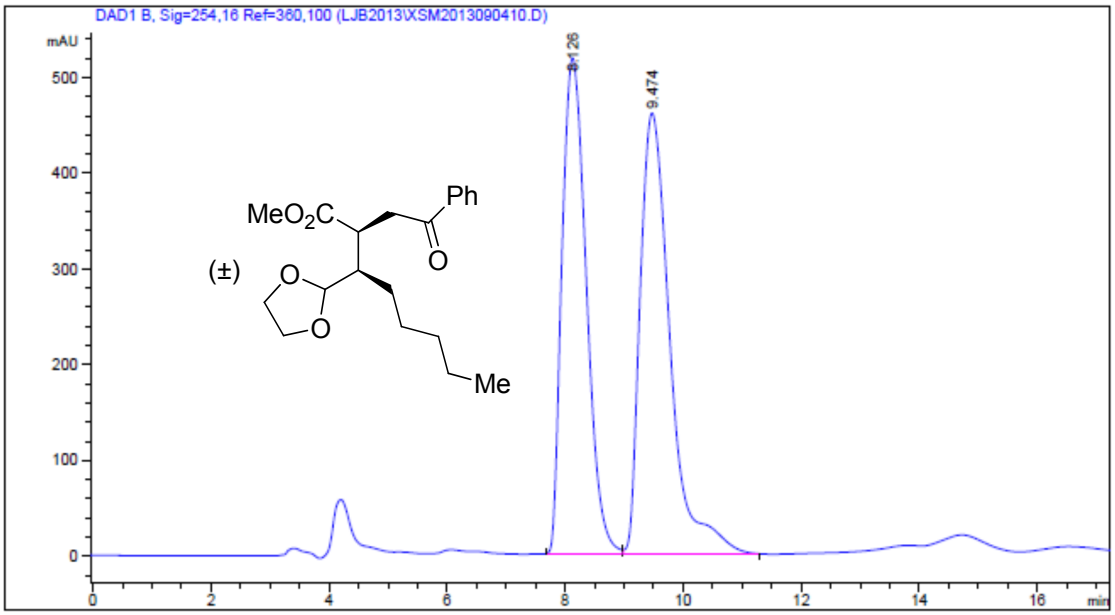



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 12.055 | 5.85057e4 | 1369.03821 | 49.17 |
| 2 | DAD 254.16 nm | 13.371 | 6.04711e4 | 1162.01135 | 50.83 |

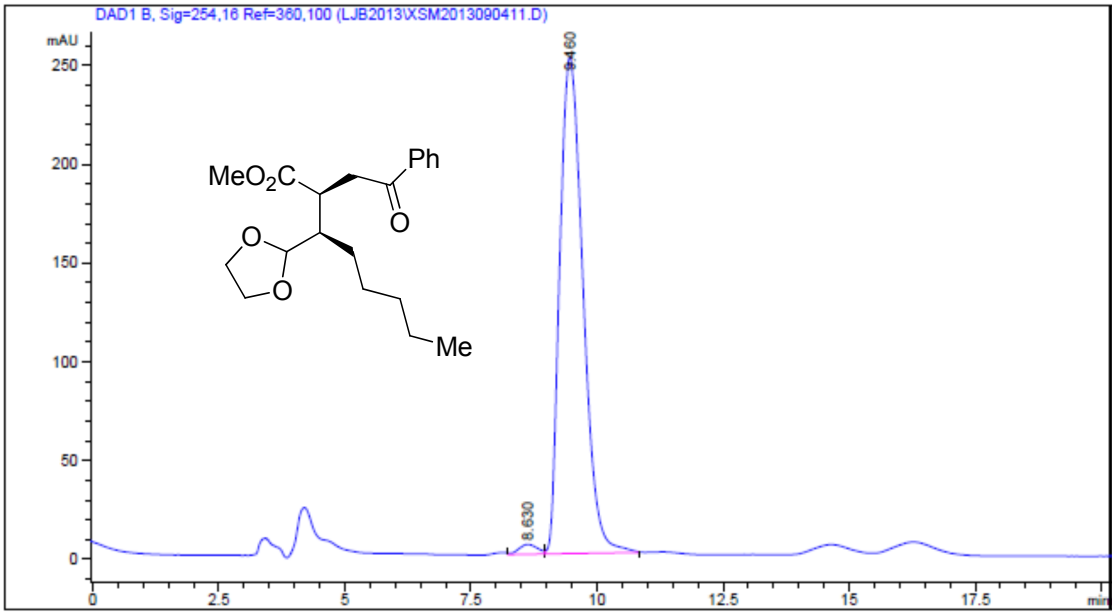



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 11.680 | 106.15650 | 3.76626 | 0.15 |
| 2 | DAD 254.16 nm | 13.108 | 7.13162e4 | 1388.42493 | 99.85 |

**4m**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

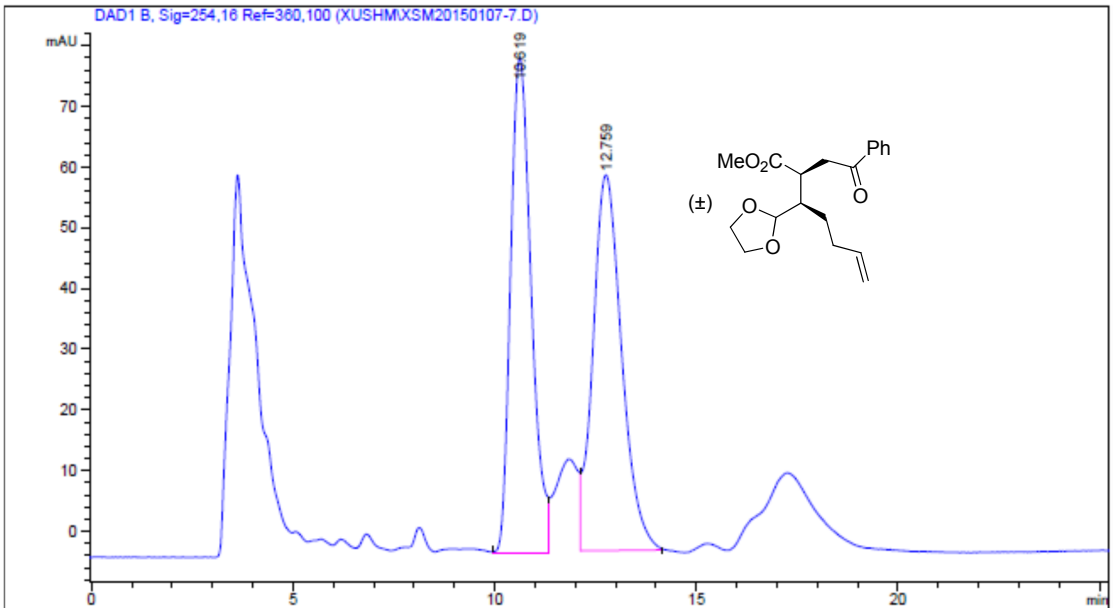



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 8.126 | 1.48138e4 | 518.13043 | 47.72 |
| 2 | DAD 254.16 nm | 9.474 | 1.62280e4 | 460.65106 | 52.28 |

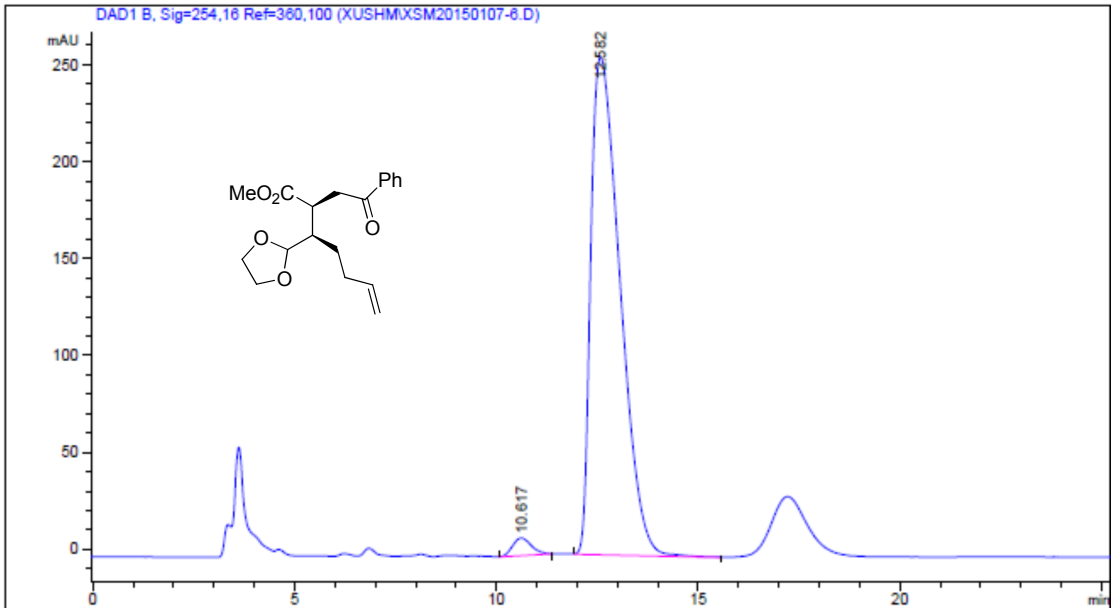



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 8.630 | 129.03970 | 5.01188 | 1.57 |
| 2 | DAD 254.16 nm | 9.460 | 8040.40137 | 251.30946 | 98.43 |

**4n**: Chiralpak AD-H Column (*n*-hexane/2-propanol = 90:10, 1.0 mL/min)

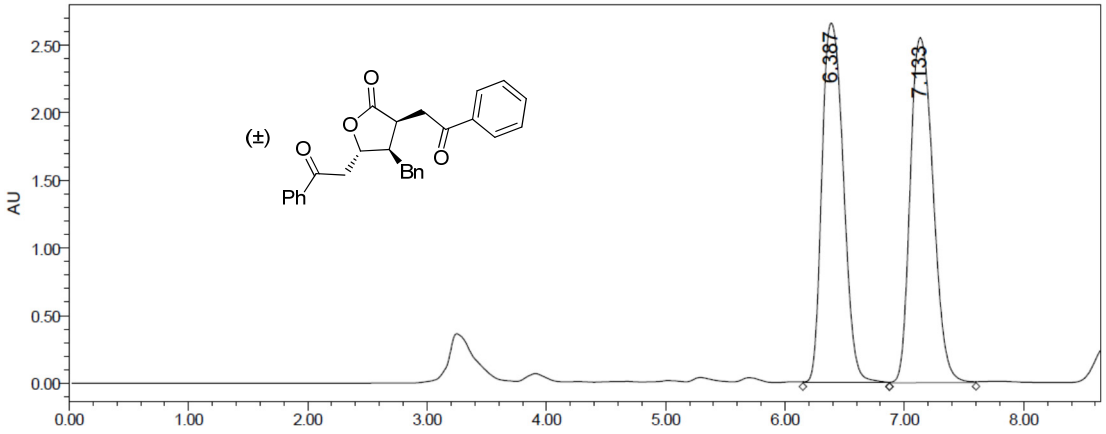



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 10.619 | 2910.53906 | 81.58125 | 48.666 |
| 2 | DAD 254.16 nm | 12.759 | 3070.10034 | 61.88708 | 51.334 |

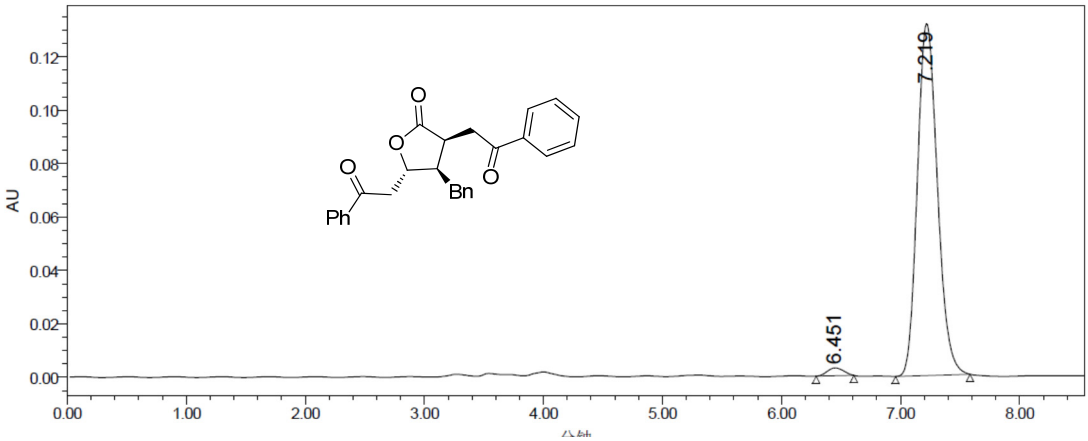



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.16 nm | 10.617 | 295.51917 | 9.07598 | 2.235 |
| 2 | DAD 254.16 nm | 12.582 | 1.29271e4 | 256.7272 | 97.765 |

**5a**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

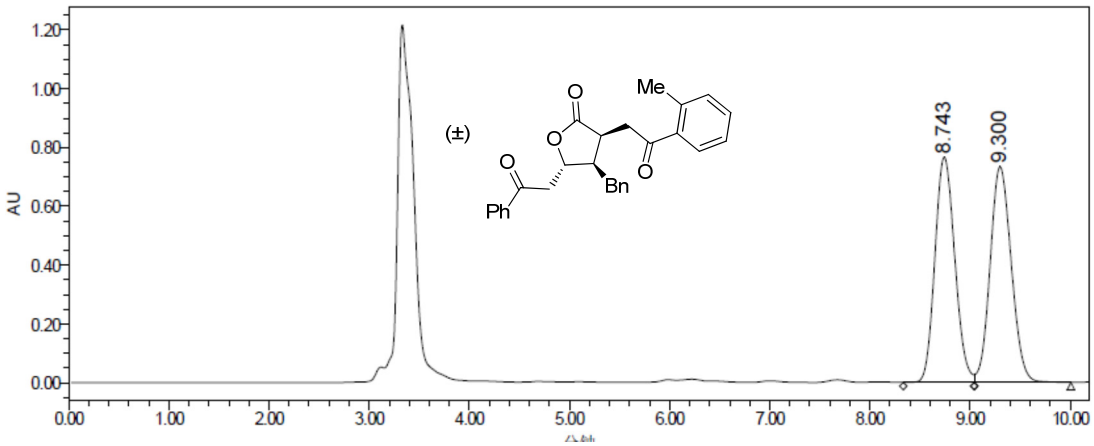



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.4 nm | 6.387 | 33229486 | 2661392 | 49.41 |
| 2 | DAD 254.4 nm | 7.133 | 34023897 | 2551202 | 50.59 |

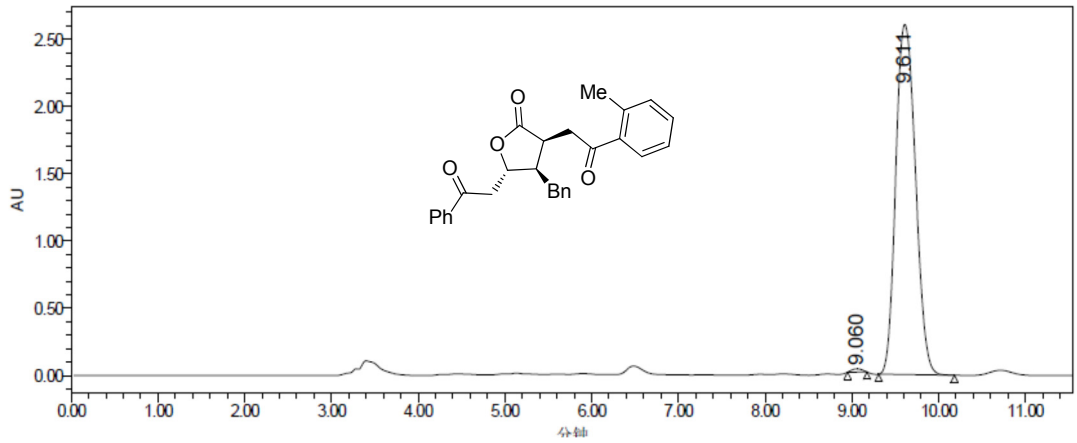



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD 254.4 nm | 6.451 | 28255 | 2946 | 1.80 |
| 2 | DAD 254.4 nm | 7.219 | 1541777 | 131804 | 98.20 |

**5b**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =60:38:2, 1.0 mL/min)

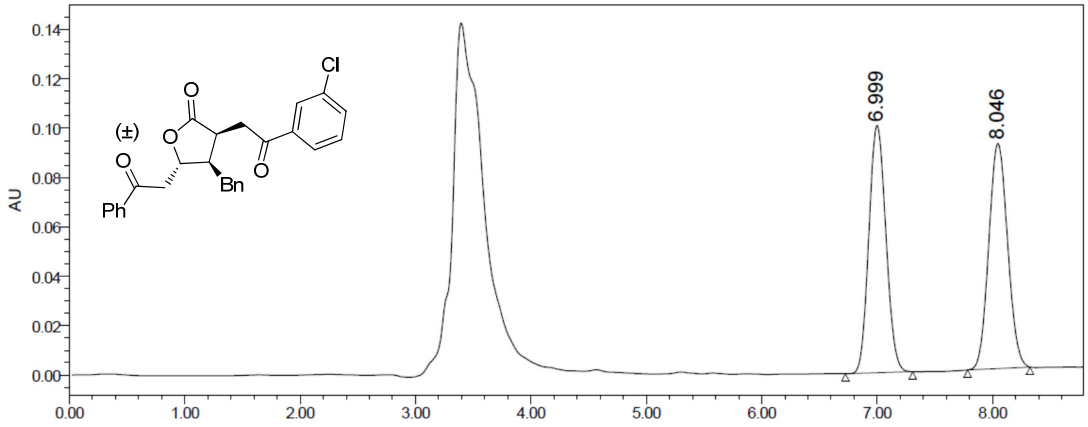



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 8.743 | 10536779 | 766283 | 49.46 |
| 2 | DAD 247.1 nm | 9.300 | 10767375 | 733451 | 50.54 |

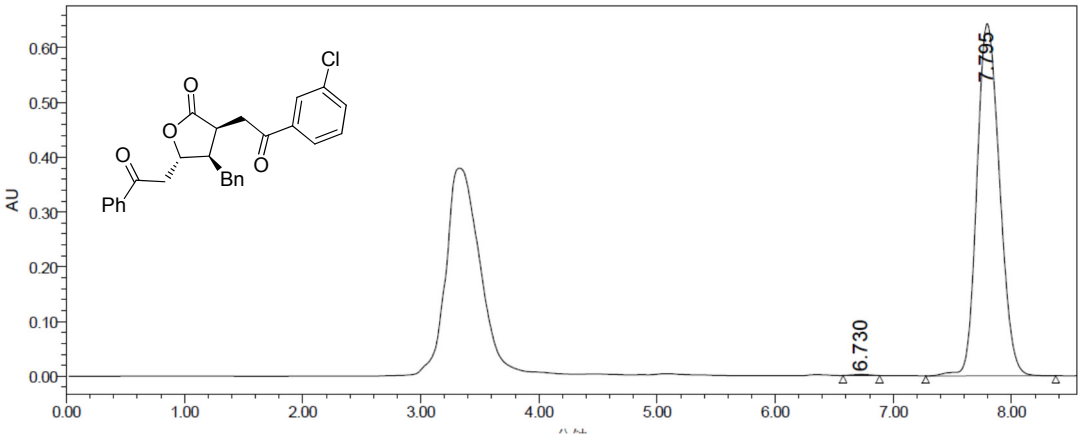



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 9.060 | 204143 | 24291 | 0.49 |
| 2 | DAD 247.1 nm | 9.611 | 41098744 | 2601465 | 99.51 |

**5c**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

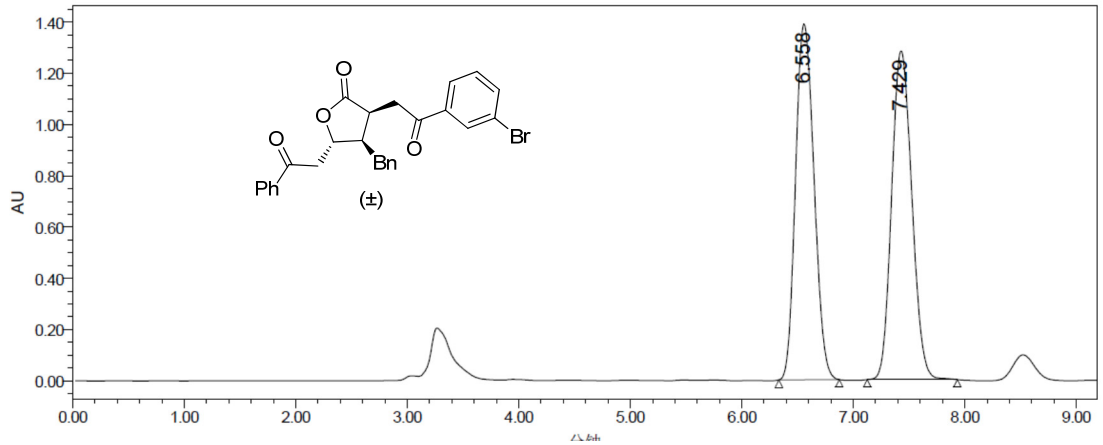



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.999 | 1029629 | 100211 | 49.90 |
| 2 | DAD 254.4 nm | 8.046 | 1033602 | 91262 | 50.10 |

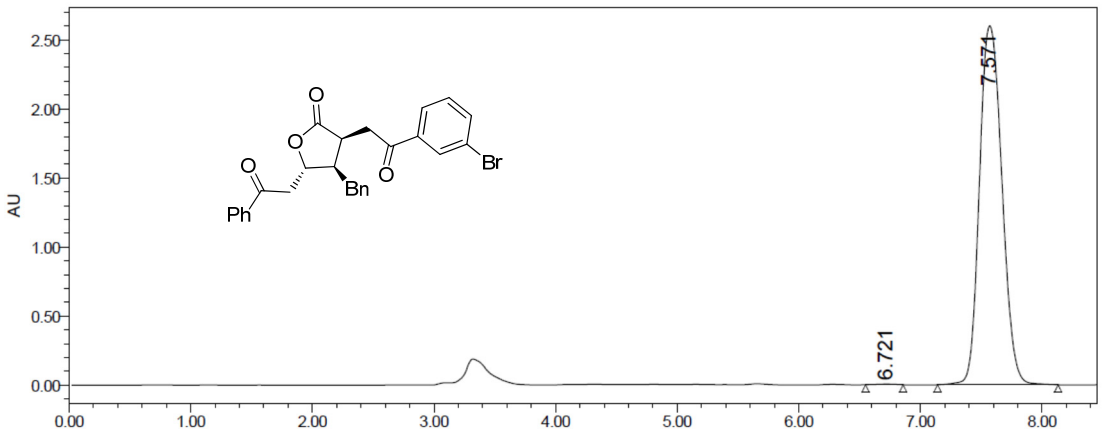



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.730 | 18289 | 1685 | 0.21 |
| 2 | DAD 254.4 nm | 7.795 | 8580794 | 643096 | 99.79 |

**5d**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

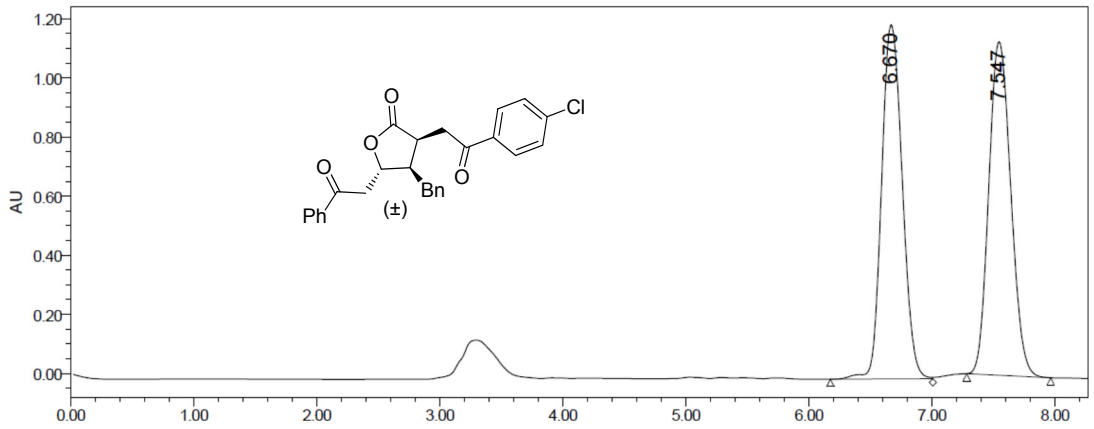



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 6.558 | 16006857 | 1388369 | 49.76 |
| 2 | DAD 247.1 nm | 7.429 | 16160231 | 1283112 | 50.24 |

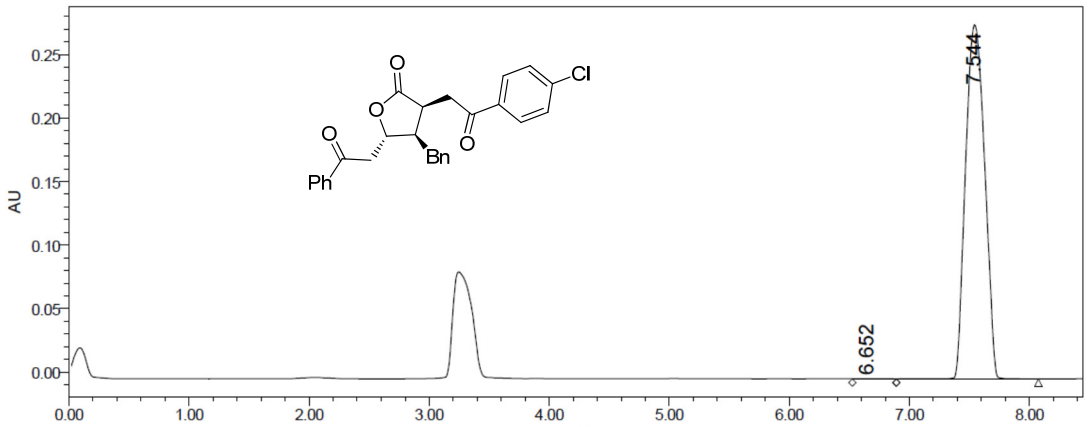



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 6.721 | 52436 | 5283 | 0.16 |
| 2 | DAD247.1 nm | 7.571 | 33424695 | 2600385 | 99.84 |

**5e**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

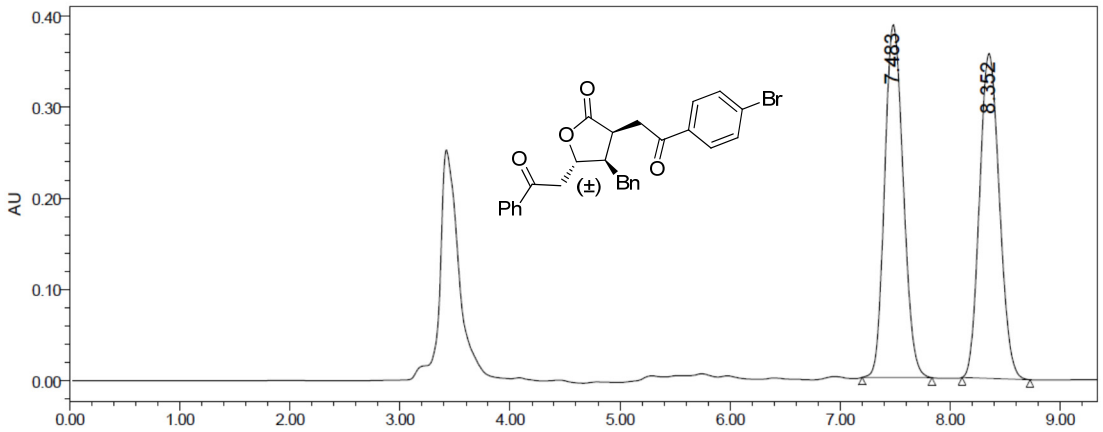



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.670 | 14005890 | 1198996 | 49.94 |
| 2 | DAD 254.4 nm | 7.547 | 14038907 | 1129518 | 50.06 |

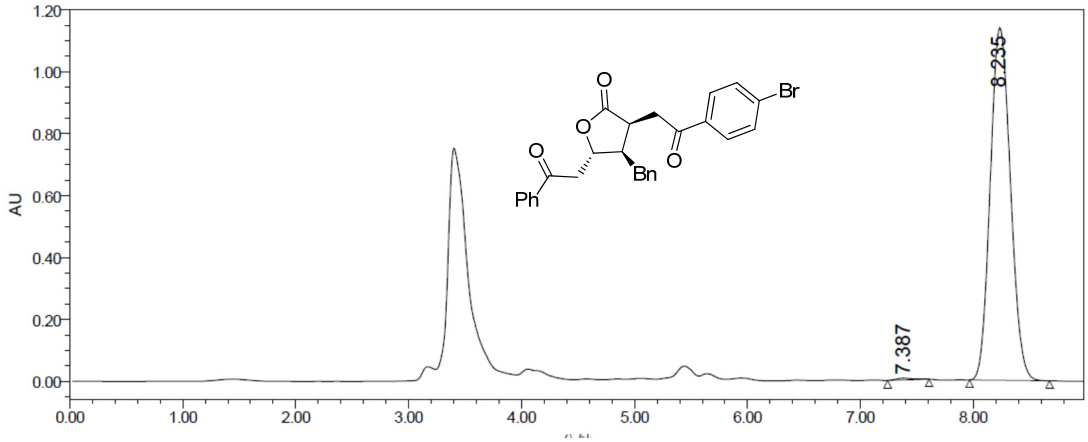



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.652 | 1433 | 119 | 0.05 |
| 2 | DAD 254.4 nm | 7.544 | 3089045 | 278861 | 99.95 |

**5f**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

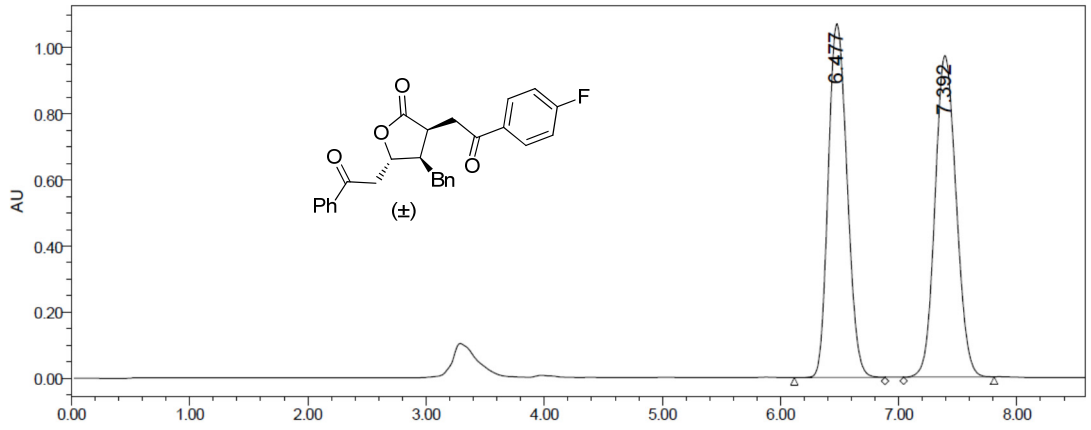



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.483 | 4552581 | 387124 | 50.49 |
| 2 | DAD247.1 nm | 8.352 | 4464947 | 356716 | 49.51 |

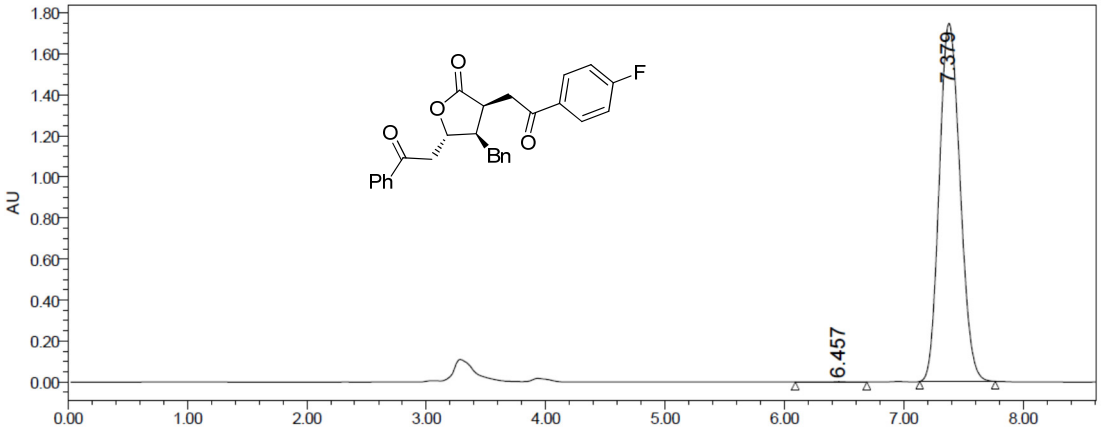



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.387 | 55759 | 5446 | 0.39 |
| 2 | DAD247.1 nm | 8.235 | 14182868 | 1138960 | 99.61 |

**5g**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

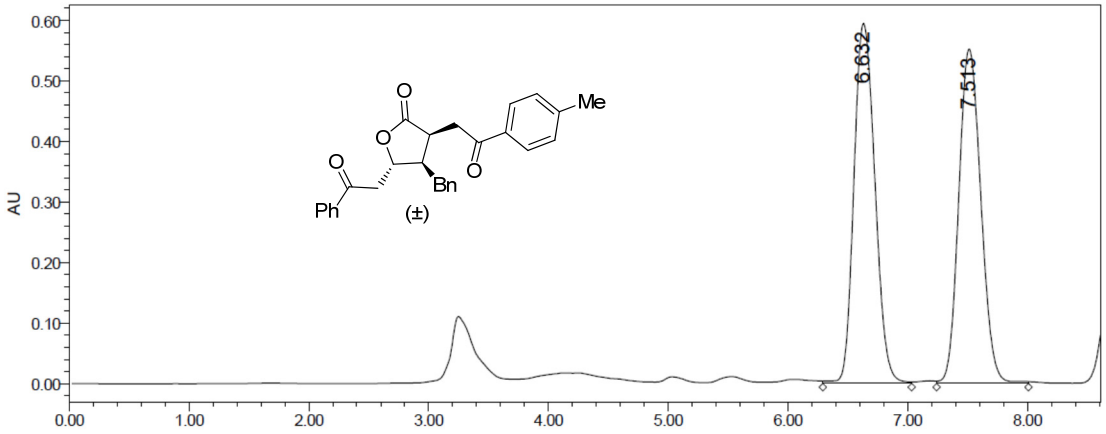



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.477 | 11873472 | 1070038 | 49.49 |
| 2 | DAD 254.4 nm | 7.392 | 12117305 | 972858 | 50.51 |

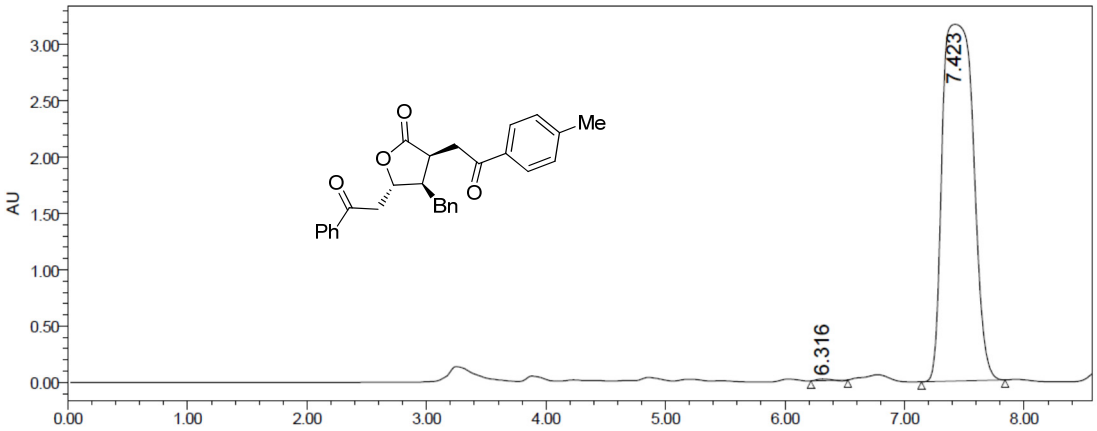



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 6.457 | 20663 | 1455 | 0.10 |
| 2 | DAD 247.1 nm | 7.379 | 21115485 | 1745849 | 99.90 |

**5h**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

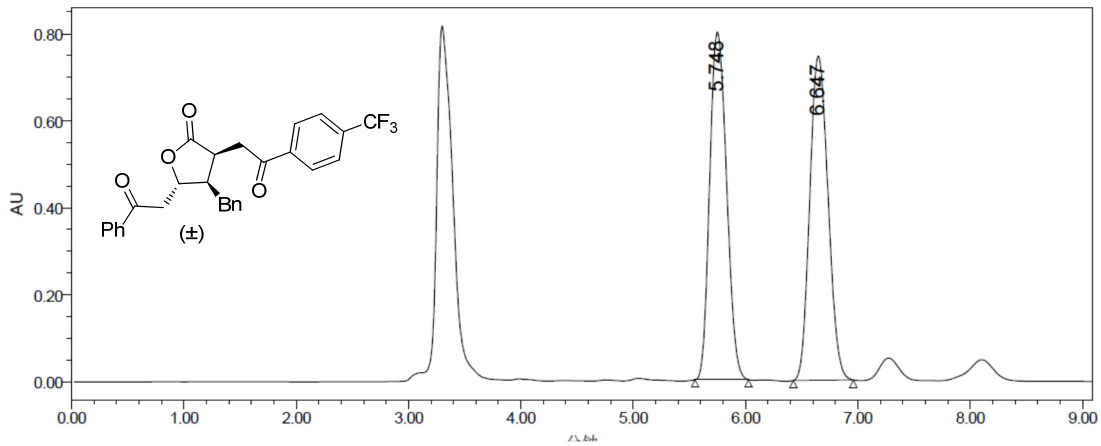



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.632 | 7250954 | 494610 | 50.57 |
| 2 | DAD 254.4 nm | 7.513 | 7086471 | 552317 | 49.43 |

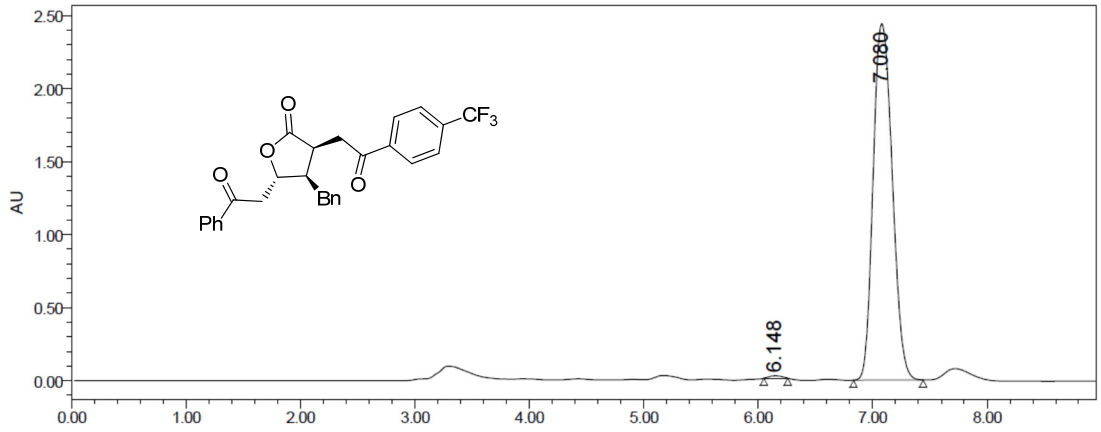



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 6.316 | 132854 | 14498 | 0.23 |
| 2 | DAD 254.4 nm | 7.423 | 57213602 | 3169253 | 99.77 |

**5i**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

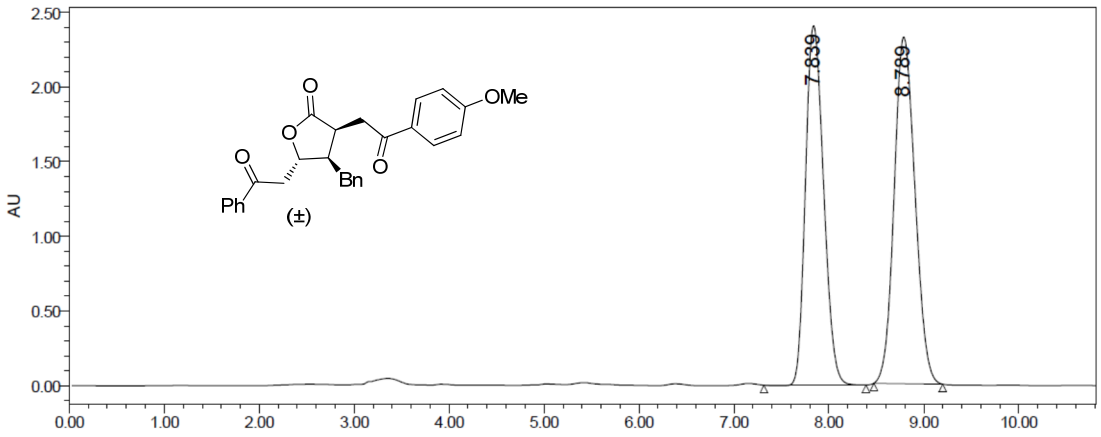



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 5.748 | 8335009 | 799335 | 49.68 |
| 2 | DAD 247.1 nm | 6.647 | 8441328 | 744817 | 50.32 |

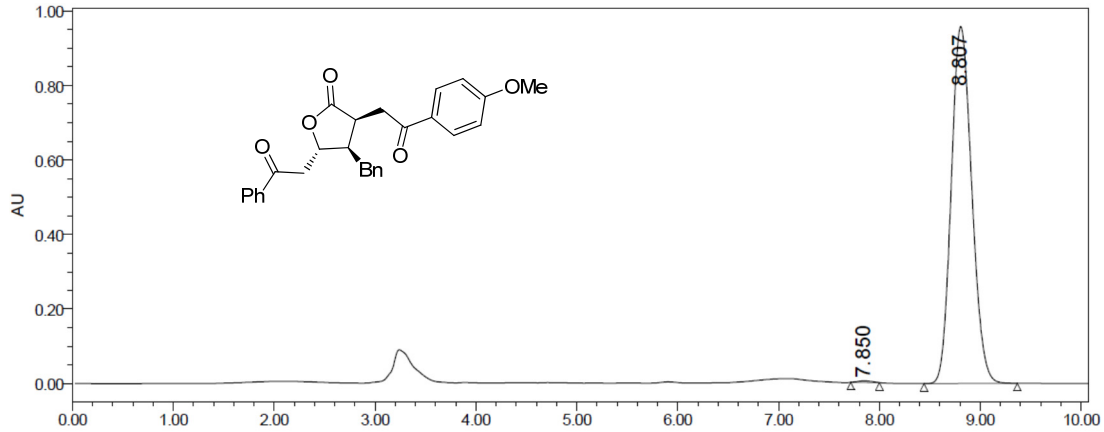



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 6.148 | 125152 | 16313 | 0.44 |
| 2 | DAD247.1 nm | 7.080 | 28367974 | 2440975 | 99.56 |

**5j**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

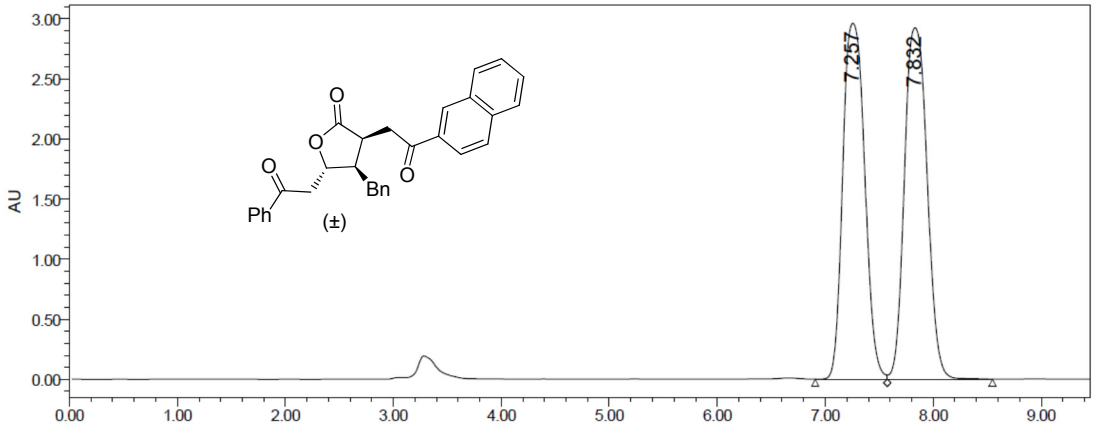



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 7.839 | 31901361 | 2405387 | 47.42 |
| 2 | DAD 254.4 nm | 8.789 | 35376414 | 2320677 | 52.58 |

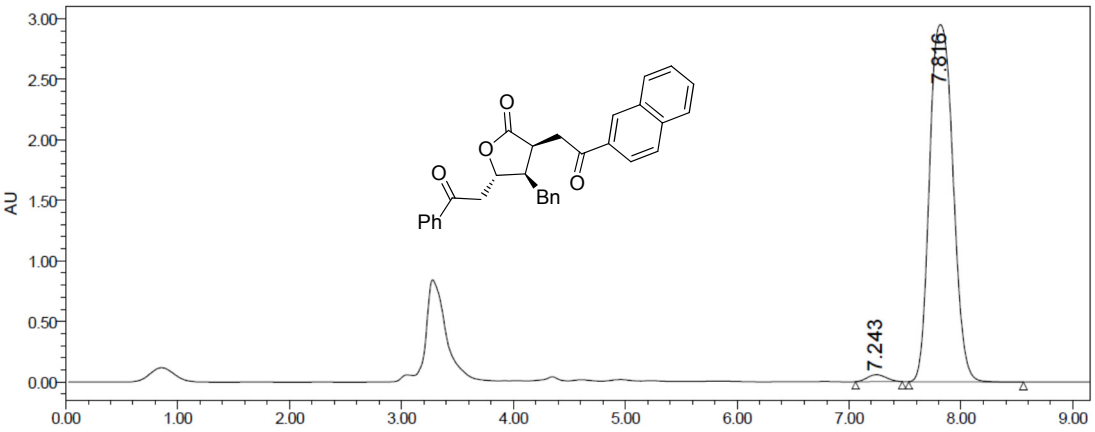



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 7.850 | 44040 | 4482 | 0.32 |
| 2 | DAD254.4 nm | 8.807 | 13525039 | 957702 | 99.68 |

**5k**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

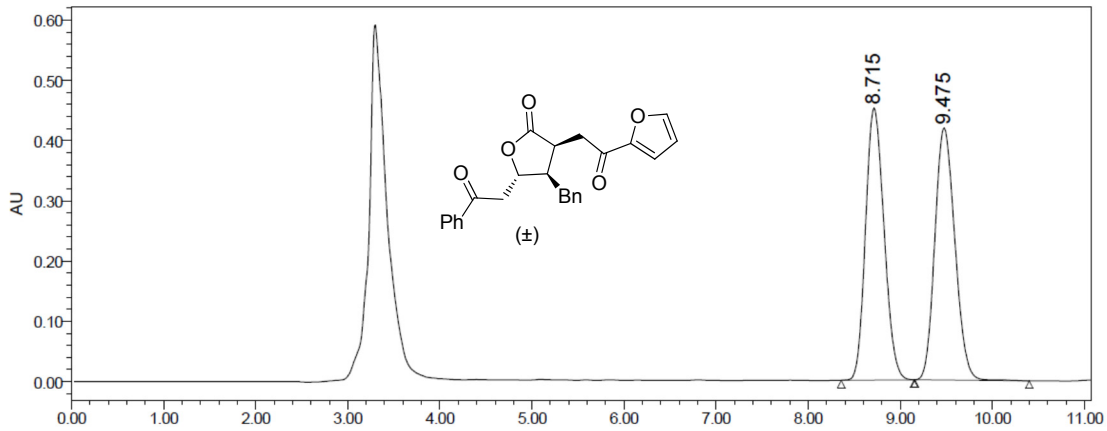



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.237 | 41613798 | 2960236 | 49.42 |
| 2 | DAD 247.1 nm | 7.832 | 42593345 | 2923344 | 50.58 |

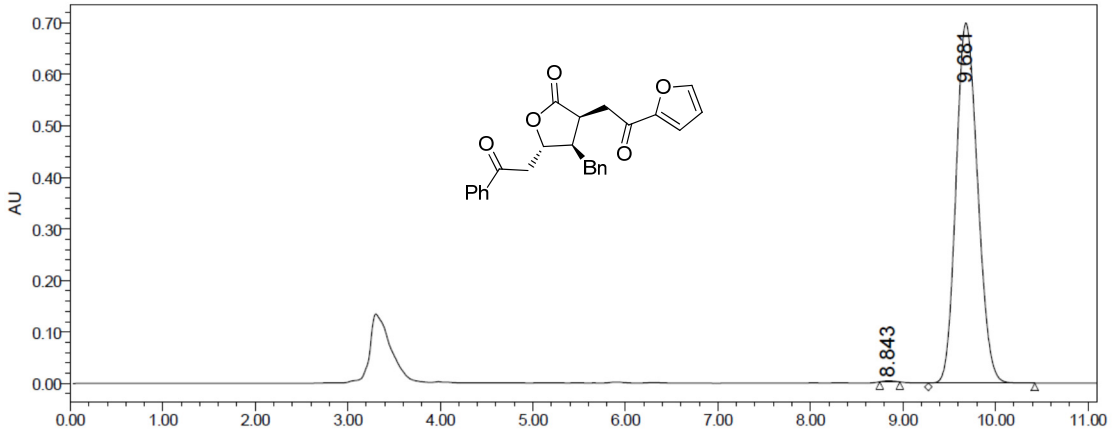



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.243 | 667312 | 56770 | 1.53 |
| 2 | DAD 247.1 nm | 7.816 | 42909348 | 2948946 | 98.47 |

**5l**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

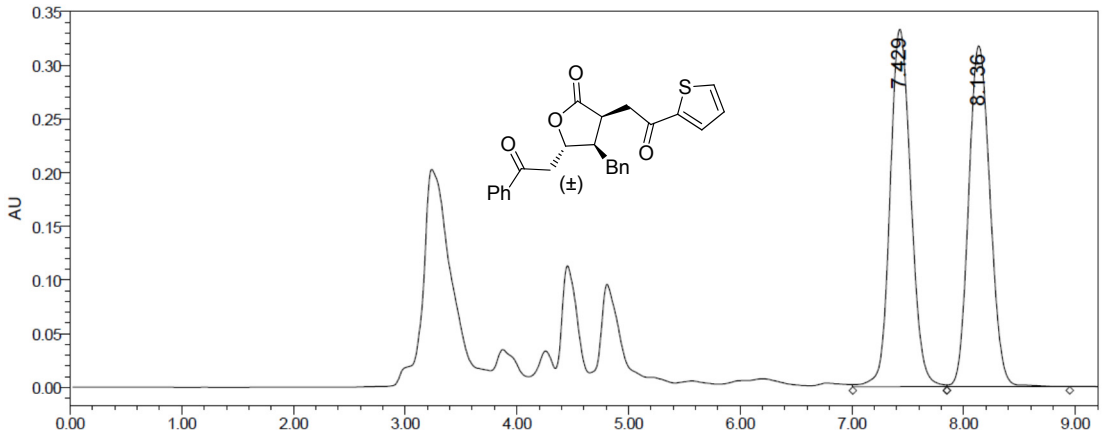



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 8.715 | 6307702 | 451341 | 49.90 |
| 2 | DAD 254.4 nm | 9.475 | 6332515 | 418446 | 51.10 |

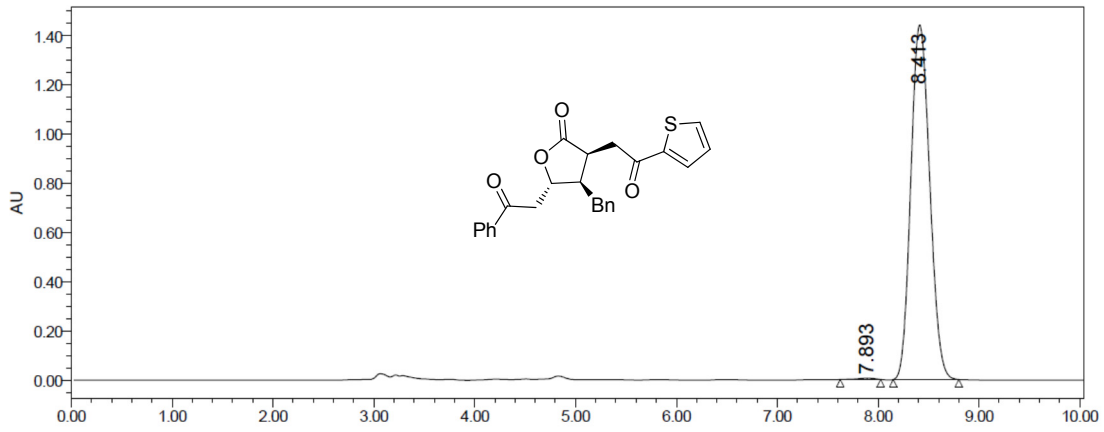



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 8.843 | 13880 | 1669 | 0.12 |
| 2 | DAD 254.4 nm | 9.681 | 11333625 | 698482 | 99.88 |

**5m**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

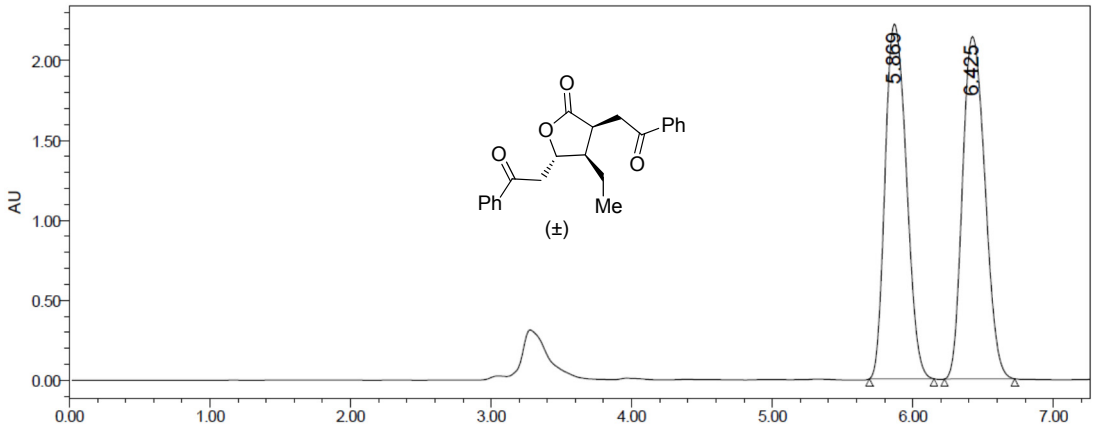



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.429 | 4322459 | 333190 | 50.55 |
| 2 | DAD 247.1 nm | 8.136 | 4228603 | 317716 | 49.45 |

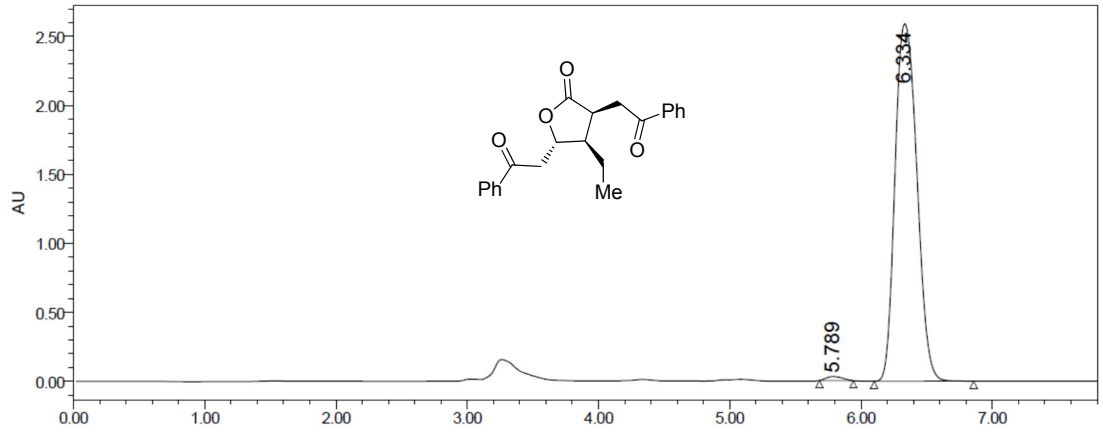



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 7.893 | 58044 | 5020 | 0.31 |
| 2 | DAD 247.1 nm | 8.413 | 18782185 | 1439624 | 99.69 |

**5n**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

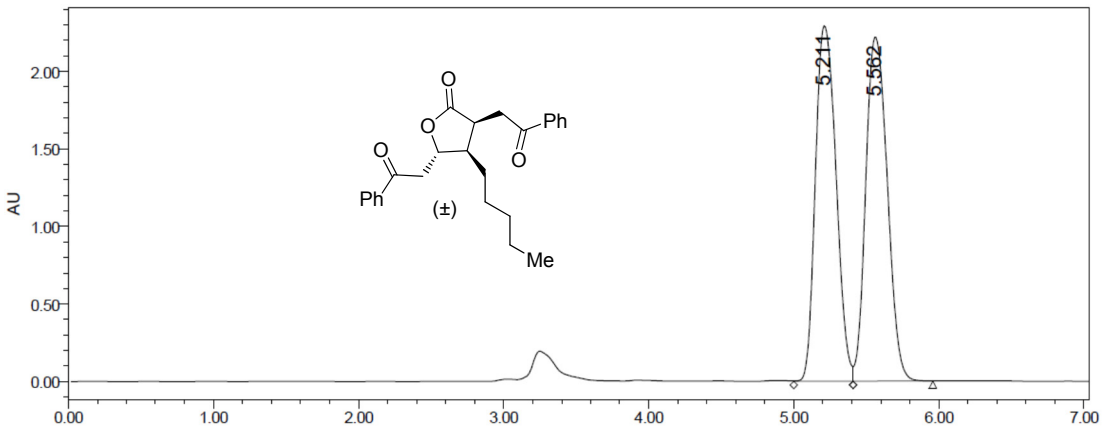



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 5.869 | 23339306 | 2219915 | 49.55 |
| 2 | DAD 247.1 nm | 6.425 | 23762240 | 2142126 | 50.45 |

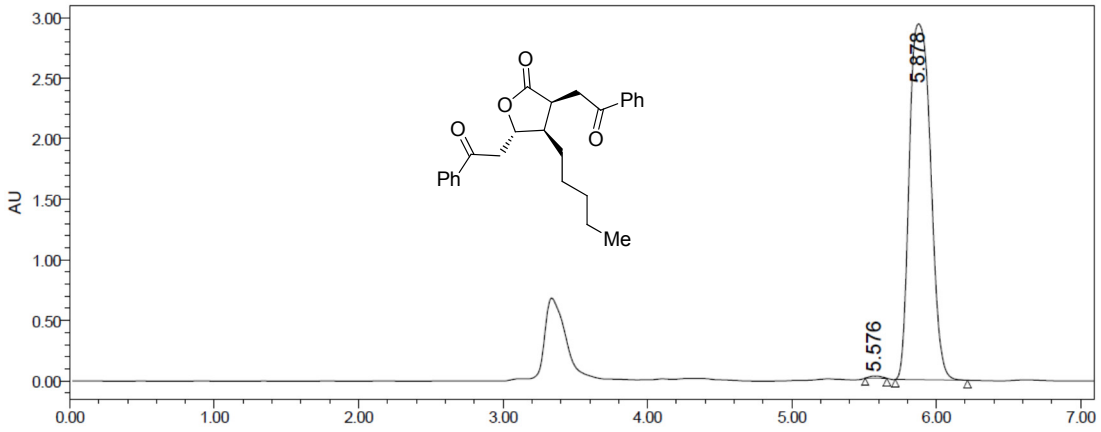



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 5.789 | 250085 | 28515 | 0.83 |
| 2 | DAD247.1 nm | 6.334 | 29995363 | 2590813 | 99.17 |

**5o**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH =50:47.5:2.5, 1.0 mL/min)

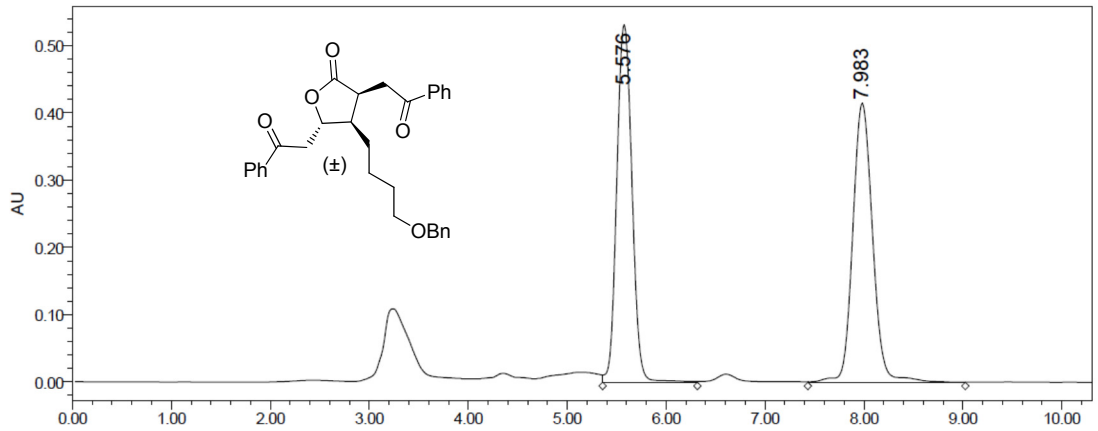



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 5.211 | 22593055 | 2292551 | 49.63 |
| 2 | DAD247.1 nm | 5.562 | 22926648 | 2221089 | 50.37 |

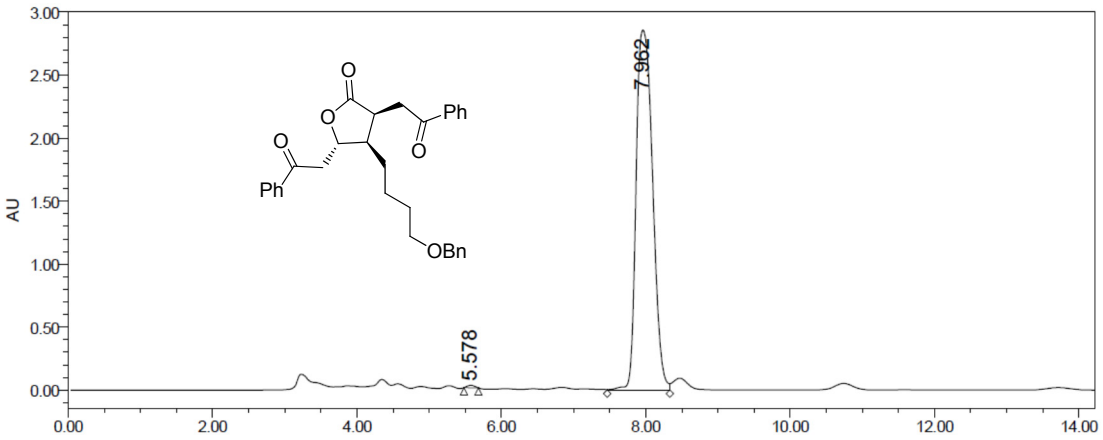



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD247.1 nm | 5.576 | 105057 | 18256 | 0.35 |
| 2 | DAD247.1 nm | 5.878 | 30190849 | 2939951 | 99.65 |

**5p**: Chiralpak IC Column (*n*-hexane/$CH_2Cl_2$/MeOH=50:47.5:2.5, 1.0 mL/min)

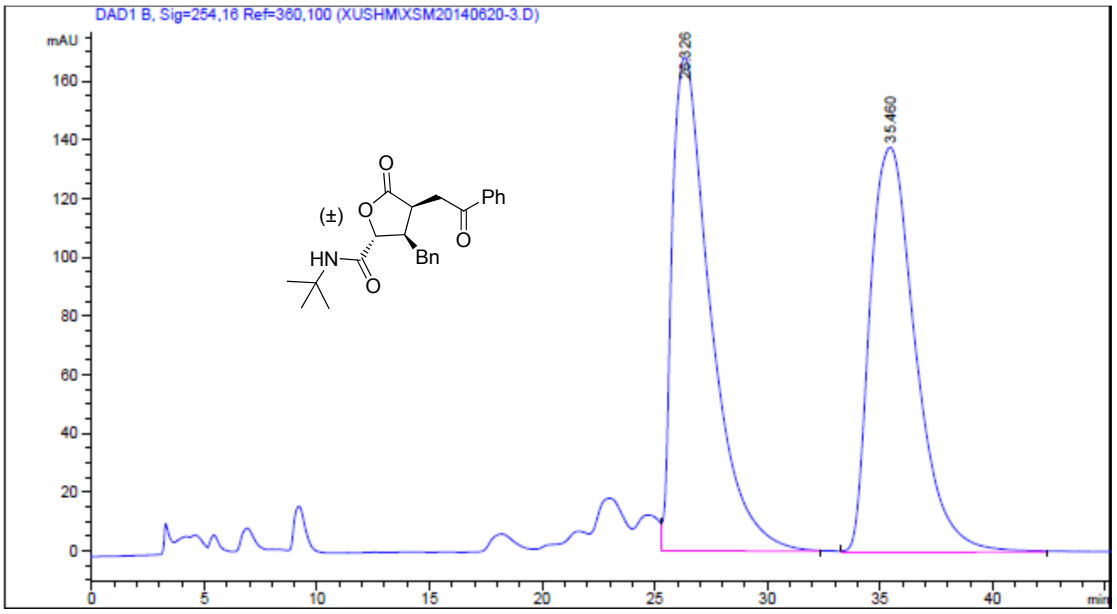



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 5.576 | 5688022 | 531468 | 49.51 |
| 2 | DAD 254.4 nm | 7.983 | 5801537 | 414828 | 50.49 |

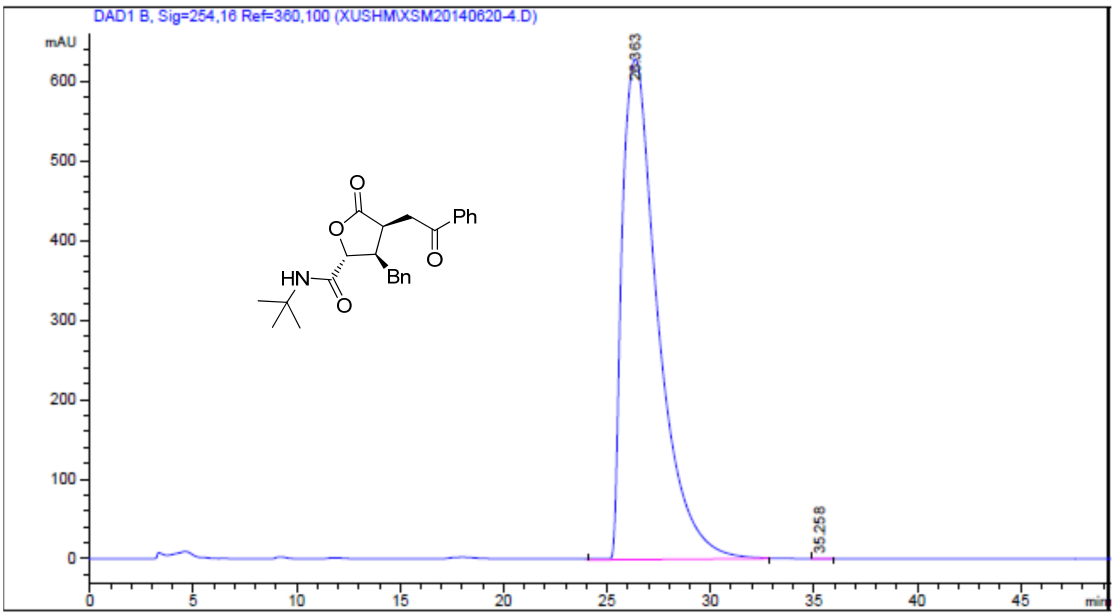



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.4 nm | 5.578 | 140301 | 19160 | 0.31 |
| 2 | DAD254.4 nm | 7.962 | 45377854 | 2856607 | 99.69 |

**6**: Chiralpak AD-H Column (*n*-hexane/2-propanol=95:5, 1.0 mL/min)

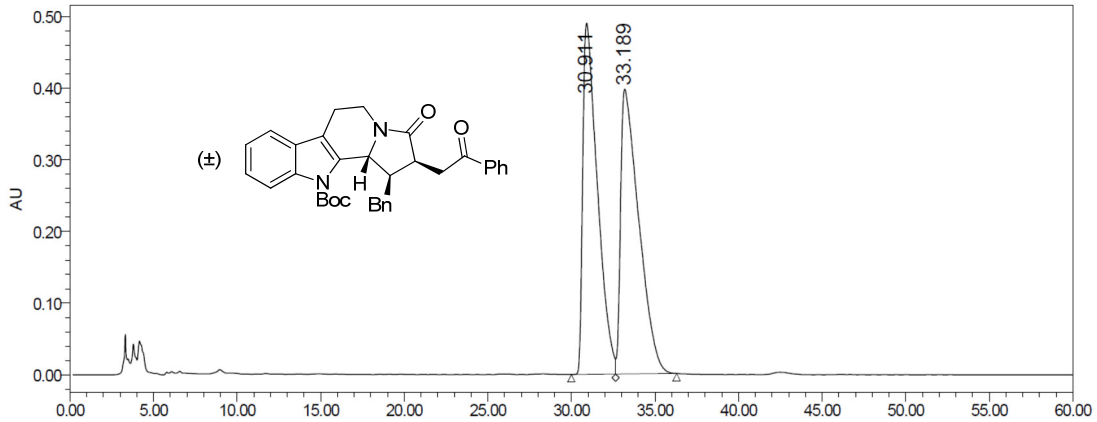



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.16 nm | 26.326 | 1.98509e4 | 168.09270 | 50.67 |
| 2 | DAD 254.16 nm | 35.460 | 1.93276e4 | 137.99440 | 49.33 |

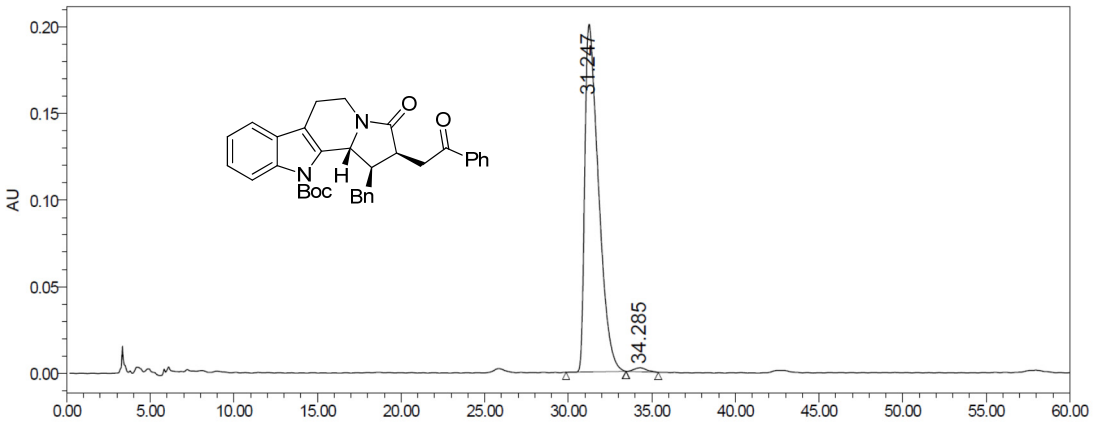



| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD254.16 nm | 26.363 | 7.53728e4 | 629.29944 | 99.99 |
| 2 | DAD 254.16 nm | 35.258 | 5.73846 | 1.54269e-1 | 0.01 |

**7**：Chiralpak ID Column (*n*-hexane/$CH_2Cl_2$/MeOH=85:14.5:0.5, 1.0 mL/min)




| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD245.0 nm | 30.911 | 29800488 | 490571 | 49.72 |
| 2 | DAD 245.0 nm | 33.189 | 30132379 | 397654 | 50.28 |




| Peak | Processed Channel | Retention Time (min) | Peak Area (mAU*s) | Peak Height (mAU) | Peak Area (%) |
|---|---|---|---|---|---|
| 1 | DAD245.0 nm | 31.247 | 11418826 | 200595 | 98.95 |
| 2 | DAD 245.0 nm | 34.285 | 120851 | 2294 | 1.05 |